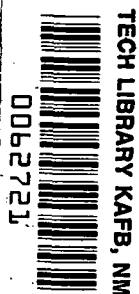


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INDUSTRIAL ION
SOURCE TECHNOLOGY

PREPARED FOR
LEWIS RESEARCH CENTER
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

GRANT 3086

Annual Report
November 1977



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16. Abstract A variety of tasks were undertaken during this grant support period. Plasma probe surveys were conducted in the 30-cm source that was designed and fabricated during the previous support period. These surveys verified that the uniformity in the ion beam is the result of a corresponding uniformity in the discharge-chamber plasma. The design, fabrication, and subsequent use of the 15-cm multipole sputtering source on grant tasks was an additional and successful application of multipole technology. The preliminary survey of texturing included several seed materials, a variety of surface materials, and both argon and tetrafluoromethane as the working gases. The texturing of stainless steel with CF ₄ appears of possible interest for medical implants. The cross section for argon-argon elastic collisions in the ion-beam energy range has been calculated from interaction potentials. This information was not previously available and permits calculation of beam interaction effects that can determine system pumping requirements. The costing section presents data that support the continued increase in ion source size. The data also indicate that different optimizations of ion-beam machines will be advantageous for long and short runs, with 1 mA-hr/cm being the rough dividing line for run length. The review of ion source technology for sputtering applications emphasizes the capacity to simultaneously optimize components in an ion-beam machine for a single application, a capacity that is not evident in competitive approaches such as diode sputtering. The development of specialized accelerator systems for specific applications will probably be a major aspect of future optimization for production applications.					
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I. INTRODUCTION

The overall objective of this grant is to further the industrial applications potential of ion-beam etching and deposition processes. To accomplish this overall objective, a variety of specific tasks have been undertaken. This report is divided into sections corresponding to the various tasks.

A 30-cm ion source designed specifically for industrial applications was described in the preceding annual report of this grant.¹ The documentation of this ion source was improved by the plasma-probe study of the discharge chamber that is reported herein.

The need for another ion source to use on etching and deposition activities of this grant led to the rapid design and fabrication of a 15-cm permanent-magnet multipole ion source. This source used technology developed previously under NASA Grants,¹⁻⁴ and is described briefly herein.

A task on ion-beam texturing was initiated during the last support period of this grant. The end objective of this task would be to develop convenient and reliable procedures for texturing a variety of surfaces. The textured surfaces could be used for functions such as the control of secondary electron emission, the increased absorption of light, and the improved attachment of biological tissue (for medical implants). A preliminary survey for this task is reported herein. This survey includes several seed materials and a variety of surface materials. Both argon and tetrafluoromethane (CF_4) were used as working gases.

A task on the scattering interaction of an argon ion beam with the argon background gas was initiated and substantially completed during this last grant period. This interaction is important to industrial

applications of ion-beam technology because it permits a large improvement in the accuracy of estimating these scattering interaction effects, thereby providing a practical basis for establishing pumping requirements for most industrial etching applications. Deposition applications may also benefit from the information of this section, but there are usually a number of other factors involved in the determination of the operating pressure required for a deposition process.

A costing task was completed during this last grant support period. The data presented will permit a rough costing estimate for etching applications. Costing estimates can also be made for deposition applications from the data presented, but at a further reduction in accuracy. Costing trends are also useful in that they can be used to indicate the future needs for work on ion-source technology.

A final section is included on the status of ion-source technology for sputtering applications. This section is essentially the same as an invited paper presented at the November 1977 meeting of the American Vacuum Society in Boston.

II. PLASMA PROBE MEASUREMENTS IN THE 30-CM DISCHARGE CHAMBER

by

Raymond S. Robinson

The discharge chamber of the 30-cm multipole ion source¹ was probed using a moveable electrostatic (Langmuir) probe to measure plasma properties as a function of position inside the discharge chamber. These plasma measurements were taken to verify the magnetic field and cathode design concepts used in the construction of the source. The use of a multipole magnetic field in the discharge chamber of the 30-cm ion source was expected to be the most important factor contributing to beam current density uniformity. This concept is an application and extension of the concepts developed by Beattie and Wilbur² using their cusped magnetic field configuration to achieve better beam uniformity. The circumferential thermionic cathode design used in the 30-cm source was also expected to contribute to beam uniformity. It was expected that uniformity in the extracted beam current density profile would be an indirect measure of the uniformity in plasma properties just upstream of the accelerator system. The multipole field, by providing a large field-free volume inside the discharge chamber should allow the plasma potential and population densities to be fairly uniform over the field-free volume.¹ The cathode was placed near the side wall of the discharge chamber to offset the decrease in plasma density usually found in that location.

Langmuir Probe

The Langmuir probe consisted of a short, cylindrical segment of 0.64 mm diameter Ta wire. The exposed portion of wire for current

collection was 2.5 mm long. The remaining length of wire was encased in alumina for insulation from the plasma. The probe was designed to move in an arc parallel to the accelerator system. The arc swept out by the probe was made to pass through the centerline of the discharge chamber. The probe could be moved through its arc from one side of the chamber to the other during source operation. The distance between the probe plane and the screen grid, however, could only be changed by adjusting the mechanical assembly between pumpdowns.

The probe potential was variable from -90V to +135V relative to the ion source body (cathode potential). The current drawn to the Langmuir probe was determined by monitoring the voltage drop across a precision sensing resistor. The probe current was displayed on an X-Y recorder as a function of probe bias potential.

Some 60 Hz interference was encountered while taking early Langmuir probe data. The first attempt to solve this problem was with a filter network at the input to the X-Y recorder. Although the quality of the traces was somewhat improved with the addition of the filter, some interference still remained. It was suspected that the cathode heater was the source of this interference because the heater voltage was about 17 V_{rms}. This heater voltage was significant compared to the 50V discharge, so that plasma fluctuations might be expected to follow the heater voltage. The cathode supply was rebuilt to give DC with 1.8 percent ripple. Using this rebuilt heater supply, no significant level of noise was encountered with the high voltage off. When the thermionic neutralizer was turned on, some noise was observed until the background pressure was reduced to 2.5×10^{-4} Torr.

A numerical analysis was performed using data of probe current versus voltage to determine plasma potential, Maxwellian electron temperature and density along with primary electron energy and density. The electron population was assumed to form two groups, a monoenergetic primary group and a lower energy Maxwellian distribution. The actual numerical technique used was a slight modification of the method of Beattie³ that facilitated somewhat more rapid convergence of the results.

Measured Plasma Properties

A survey of plasma properties was taken with the Langmuir probe located in the center of the discharge chamber. Figure 2-1 shows the nearly linear increase of Maxwellian electron density as the discharge current is increased. Because the discharge potential is held constant, the abscissa is proportional to total discharge power. Thus, the expected linear increase of electron density with power at constant voltage is observed.⁴

With the Langmuir probe again located in the center of the discharge chamber, various plasma properties were measured as a function of discharge potential. As the discharge potential was raised from 45V to 95V, the discharge current was reduced from 1.5 to 1.1 amps to maintain a constant beam current of 300 ma. The background pressure was held to a nominal 2.5×10^{-4} Torr with a few variations of $+0.1 \times 10^{-4}$ and -0.2×10^{-4} Torr.

Figure 2-2 shows the measured primary and Maxwellian electron densities as a function of discharge potential along with the temperature of the Maxwellian distribution. For practical purposes, the total plasma density can be taken as just the density of the Maxwellian

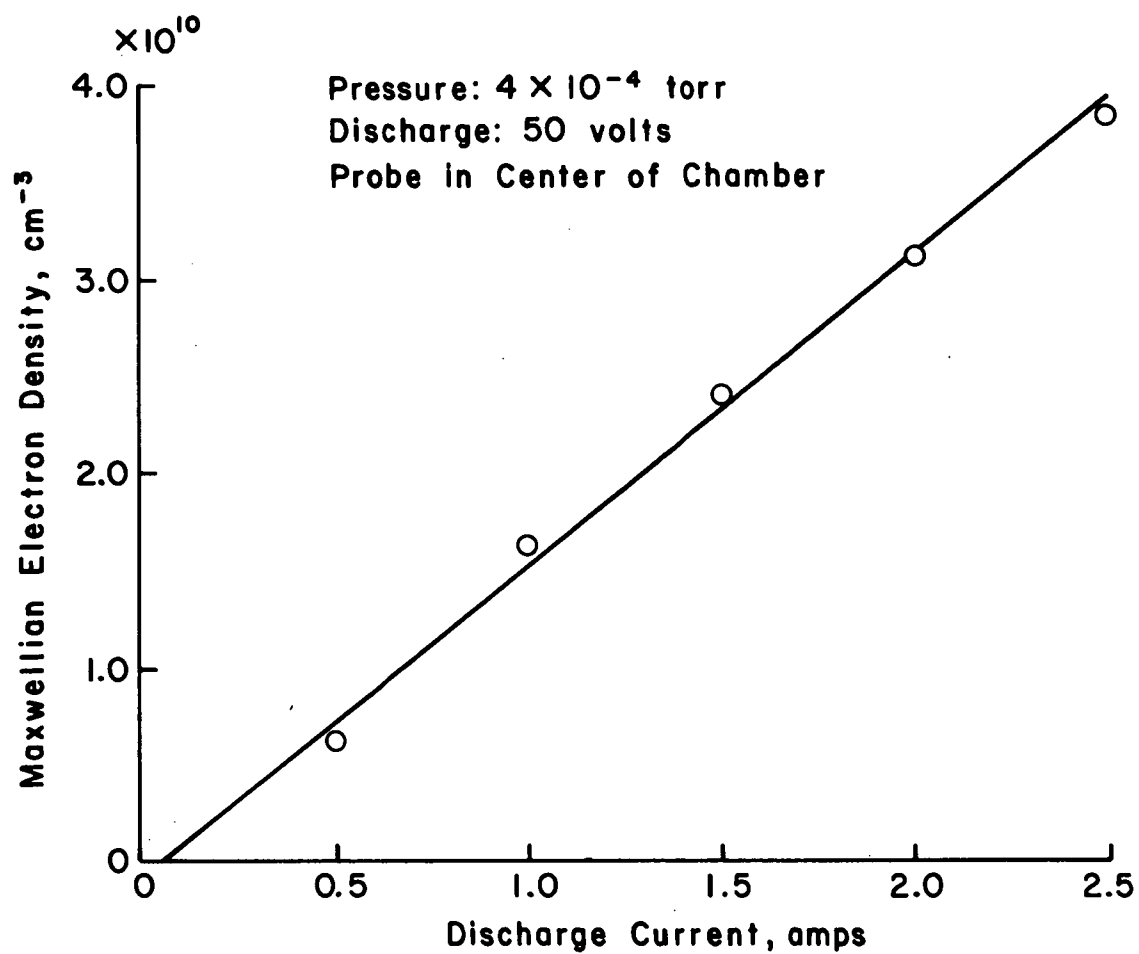


Figure 2-1. Maxwellian electron density at the center of the discharge chamber as a function of discharge current.

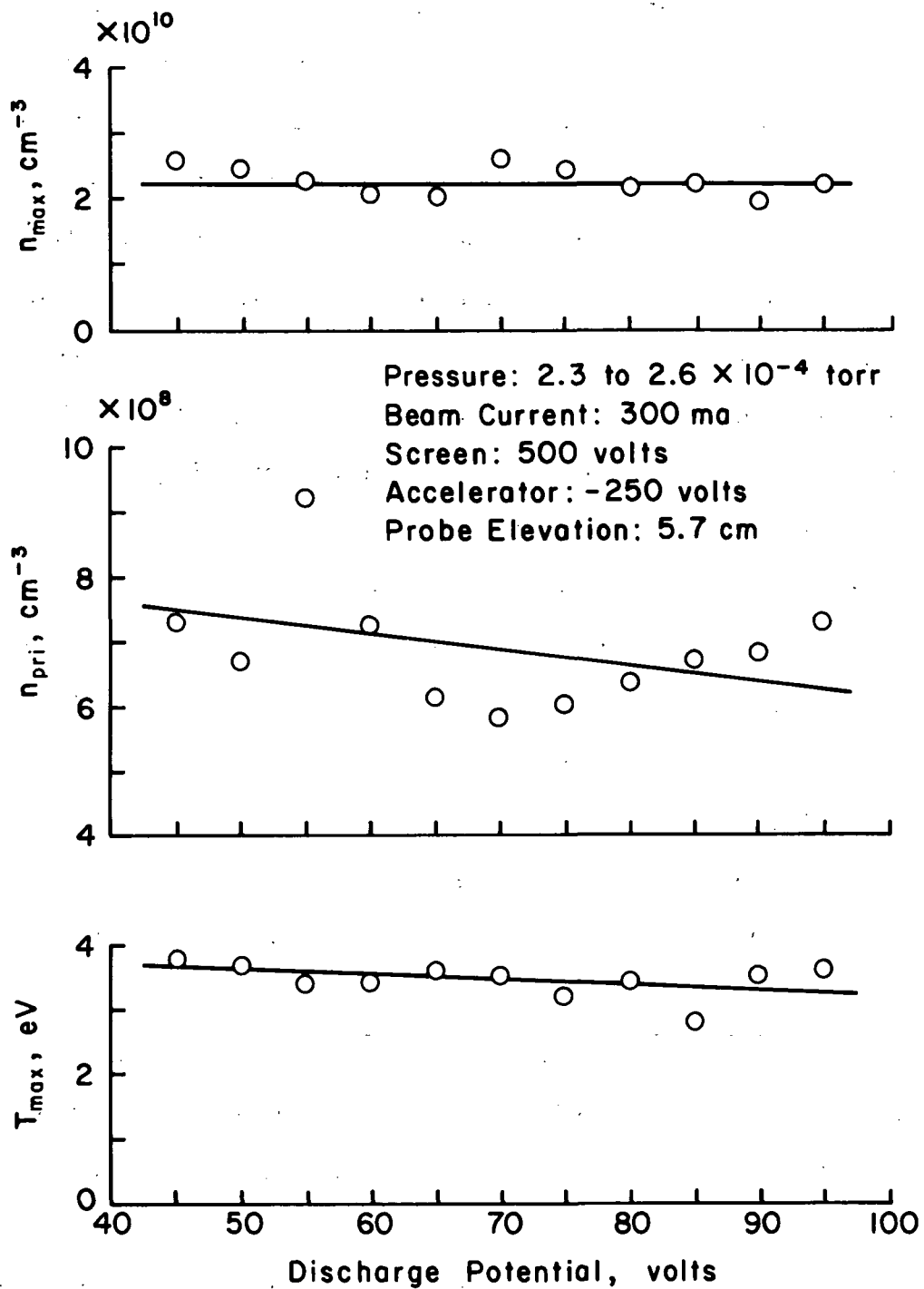


Figure 2-2. Measured primary and Maxwellian electron densities and Maxwellian temperature as a function of discharge potential.

electrons. The Maxwellian density is very nearly constant over this wide range of discharge potential. This follows from the requirement that the beam current, which is extracted directly from the plasma, remain constant. The discharge power, however, clearly increases by more than 50 percent without a corresponding increase in Maxwellian density such as that observed in Fig. 2-1. It is probable that, in this case, the higher discharge voltages make accessible more excitations and multiple ionizations of the working gas which would account for the additional power required to maintain a constant plasma density and, therefore, a constant beam current. Double ionization, particularly, is seen to increase sharply with discharge potential as shown by the ion production rate factors calculated by Wilbur.⁵

The measured primary electron density is typically found to be down by about a factor of 30 from the Maxwellian density. This is somewhat larger than the 10 to 1 ratio of Maxwellian to primary electrons typical of a smaller, 15-cm multipole chamber operated on argon.⁶

The temperature of the Maxwellian distribution is approximately constant as the discharge potential is increased by more than a factor of two and the discharge power is increased by more than 50 percent. The temperature of the Maxwellian distribution is probably governed more strongly by the energies of the lowest level excitations through which the electrons can readily give up energy than by bulk conduction or plasma properties.

Figure 2-3 shows the measured plasma potential and primary electron energy as a function of discharge potential. The plasma potential exhibits a linear increase with discharge potential, with a slope greater than unity. It should be pointed out that the plasma density

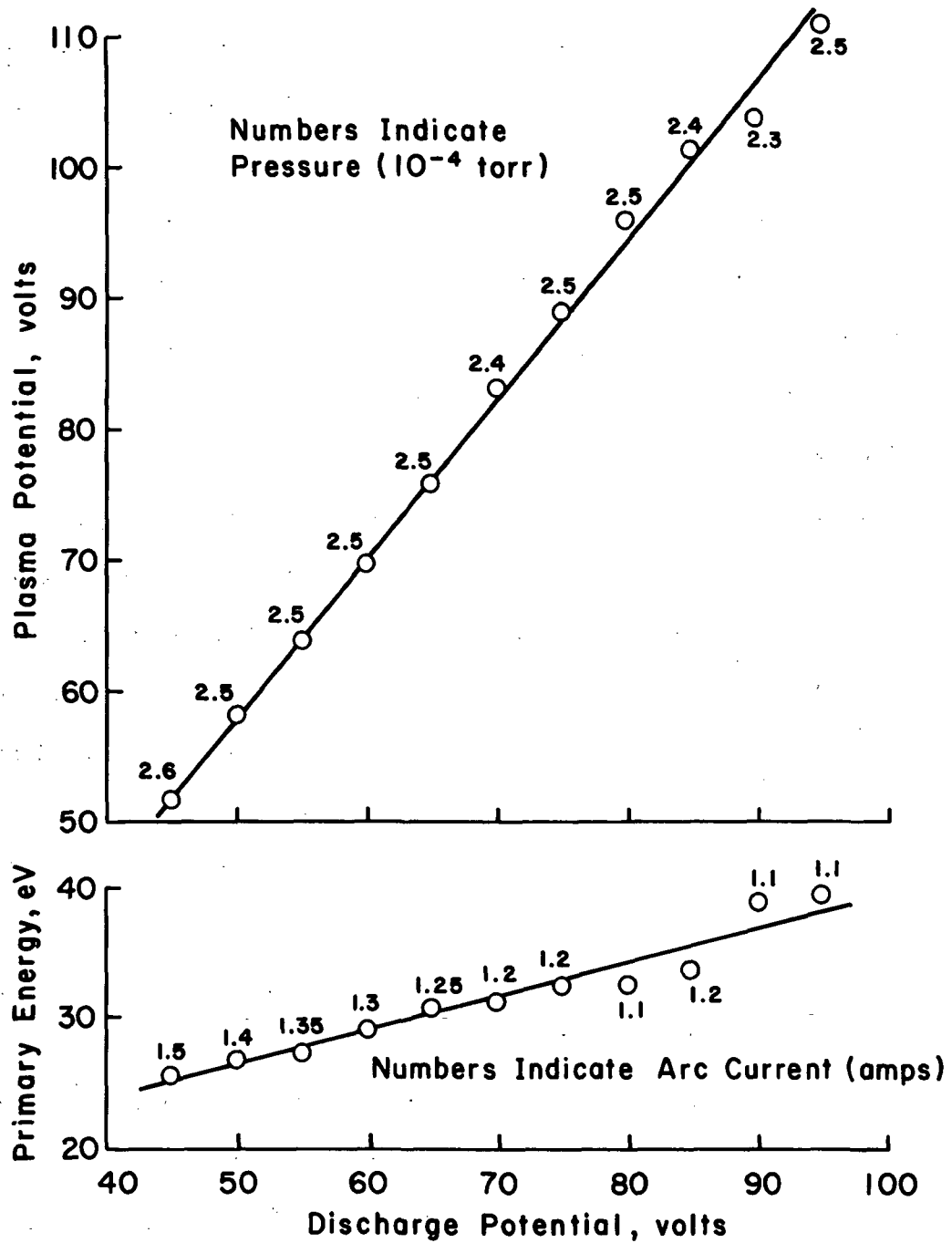


Figure 2-3. Measured plasma potential and primary electron energy as a function of discharge potential.

and Maxwellian temperature are held constant as the discharge potential is varied. Thus the properties of the plasma in the vicinity of the anodes are approximately the same over the entire range in discharge potential from 45V to 95V. Because the discharge current must decrease from 1.5 amps to 1.1 amps to maintain the constant plasma properties, it follows that the voltage between the anode and the plasma must increase to reduce the collection of discharge current at the anodes. It is this additional increase in plasma potential beyond the increase in discharge potential that would account for a slope greater than unity.

The primary electron energy shows a considerably lower value and slower rate of increase than might be expected from the simple two-population model assumed in the analysis of the Langmuir probe data.³ The calculated primary energies range from about 57% of the discharge potential down to about 42% of the discharge potential as the discharge potential increases from 45V to 95V. The absence of a clearly defined monoenergetic population of electrons at approximately the discharge energy is possibly a result of an enhanced secondary electron production at energies intermediate between the Maxwellian temperature and the discharge potential. A spread-out distribution of higher energy electrons could yield as the indicated primary energy, an approximate average of the energies of these higher energy electrons when the two population analysis procedure is used to reduce Langmuir probe data.

At fixed source operating conditions (see Fig. 2-4), Langmuir probe measurements were taken as a function of radial distance from the center of the discharge chamber in a plane located parallel to and 5.7 cm above the accelerator system. Figure 2-4 is a plot of primary and Maxwellian electron densities as a function of radius from the center of the chamber

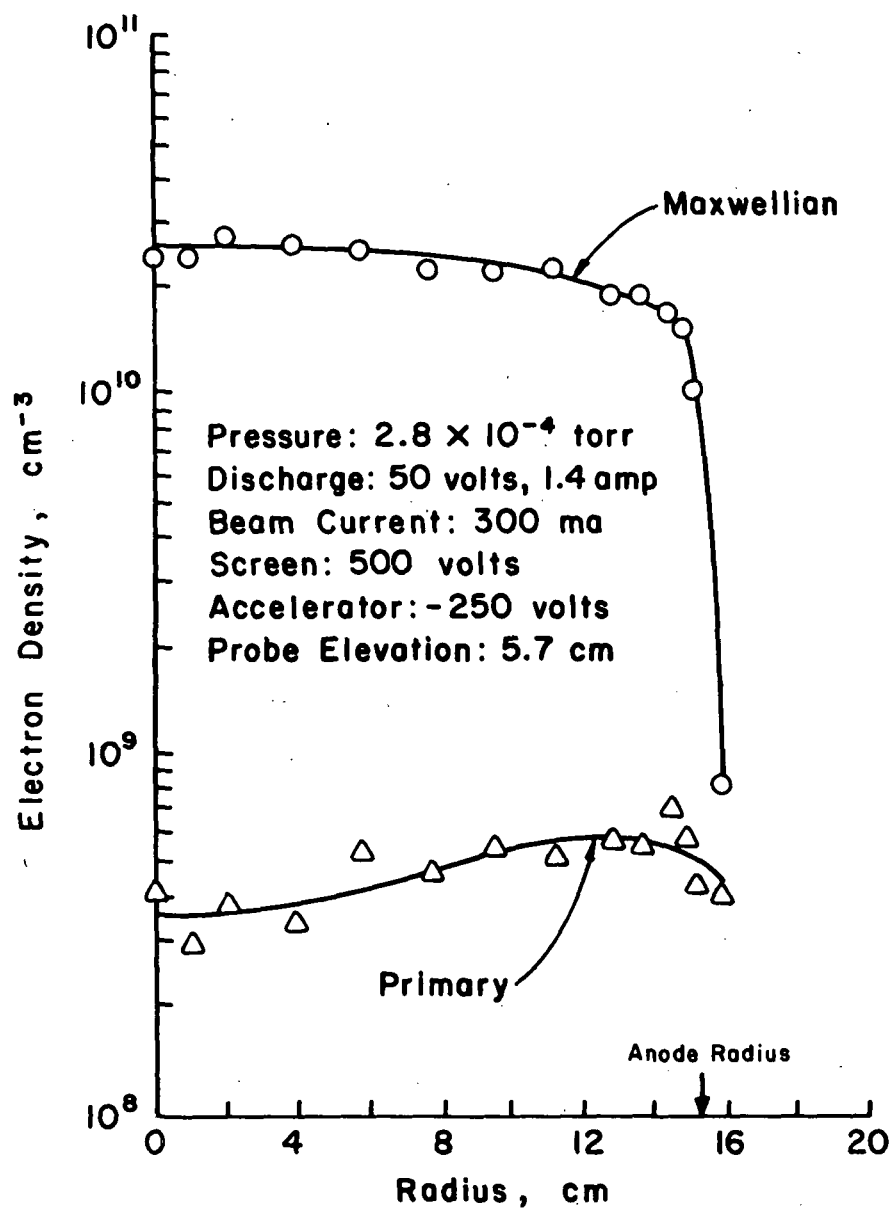


Figure 2-4. Measured plasma potential, primary energy and Maxwellian temperature as a function of radius in a plane 5.7 cm from the screen grid.

out to the anode radius and slightly beyond. The plasma density exhibits only a slow decrease with radius over most of the field-free region but an abrupt drop of more than a factor of 10 is observed within a distance of a little more than 1 cm at the position of the anode. The plasma in the field-free volume appears to be well-contained with little variation in density, an indication that the chosen multipole field configuration is adequate for the desired electron containment.

The measured primary electron density, while considerably lower than the Maxwellian electron density, shows an important enhancement in population near the location of the thermionic cathode. This is a clear result of cathode location, emphasizing the importance of the circumferential cathode for maintaining ion generation and plasma density at these peripheral locations.

Figure 2-5 shows the measured plasma potential, the primary electron energy, and the Maxwellian temperature as a function of radius from the center of the discharge chamber. The data are again taken in a plane 5.7 cm from the accelerator system. The plasma potential is nearly uniform over most of the radius, as might be expected from the near uniform plasma density and negligible magnetic field. Primary energy rises slightly as the radius is increased, reaching a maximum at about 60 percent of the maximum radius, i.e. near the cathode location. The observed minimum in primary electron energy along the chamber axis is probably also a result of the circumferential cathode configuration. Both plasma potential and primary electron energy show a sharp dip at about anode radius. The Maxwellian temperature is, as closely as can be determined, uniform over the ion source radius.

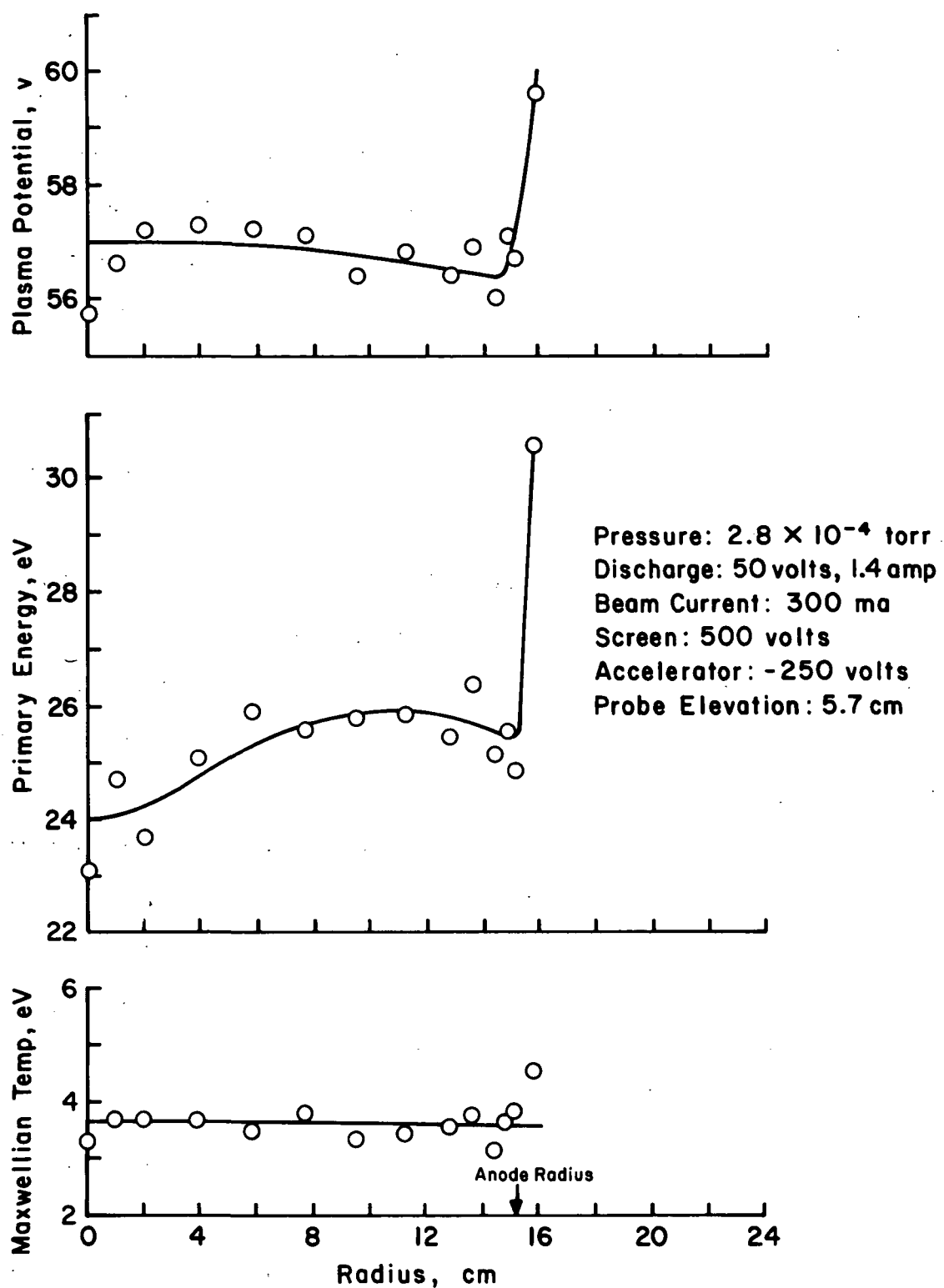


Figure 2-5. Measured plasma potential, primary energy and Maxwellian temperature as a function of radius in a plane 5.7 cm from the screen grid.

Figure 2-6 is a survey of the same plasma parameters shown in Fig. 2-5 except the data now reflect properties in a plane about 1 cm above the accelerator system. The dips in plasma potential and primary energy near the anode radius are much more pronounced closer to the accelerator system. Both the primary energy and the Maxwellian temperature show similar qualitative behavior to the curves taken farther from the accelerator system. The plasma potential is down slightly while both the primary energy and Maxwellian temperature show small increases. The Maxwellian temperature remains a constant across the radius of the discharge chamber.

Figure 2-7 shows the densities of primary and Maxwellian electrons measured just upstream of the accelerator system (~ 1 cm). These curves are also qualitatively similar to those taken farther from the accelerator system, although the primary density at this distance from the cathode no longer exhibits a lobe of enhanced density.

A comparison between the plasma density shown in Fig. 2-7 and the extracted beam ion current density profiles shown in Fig. 2-8 shows good qualitative agreement; the flatness of the beam current density profile appears to reflect the flatness in the plasma density near the grids.

The design concepts of a multipole discharge chamber, allowing a large, central, field-free volume along with the placement of a circumferential thermionic cathode, are amply supported by the measured plasma properties and by the current density profile in the beam extracted from this plasma. The multipole concept can be expected to provide plasma containment volumes of virtually any size or shape, thereby allowing ion beam cross-sections to be tailored to the needs of specific applications areas such as ion beam etching, surface texturing or sputter deposition of thin films.

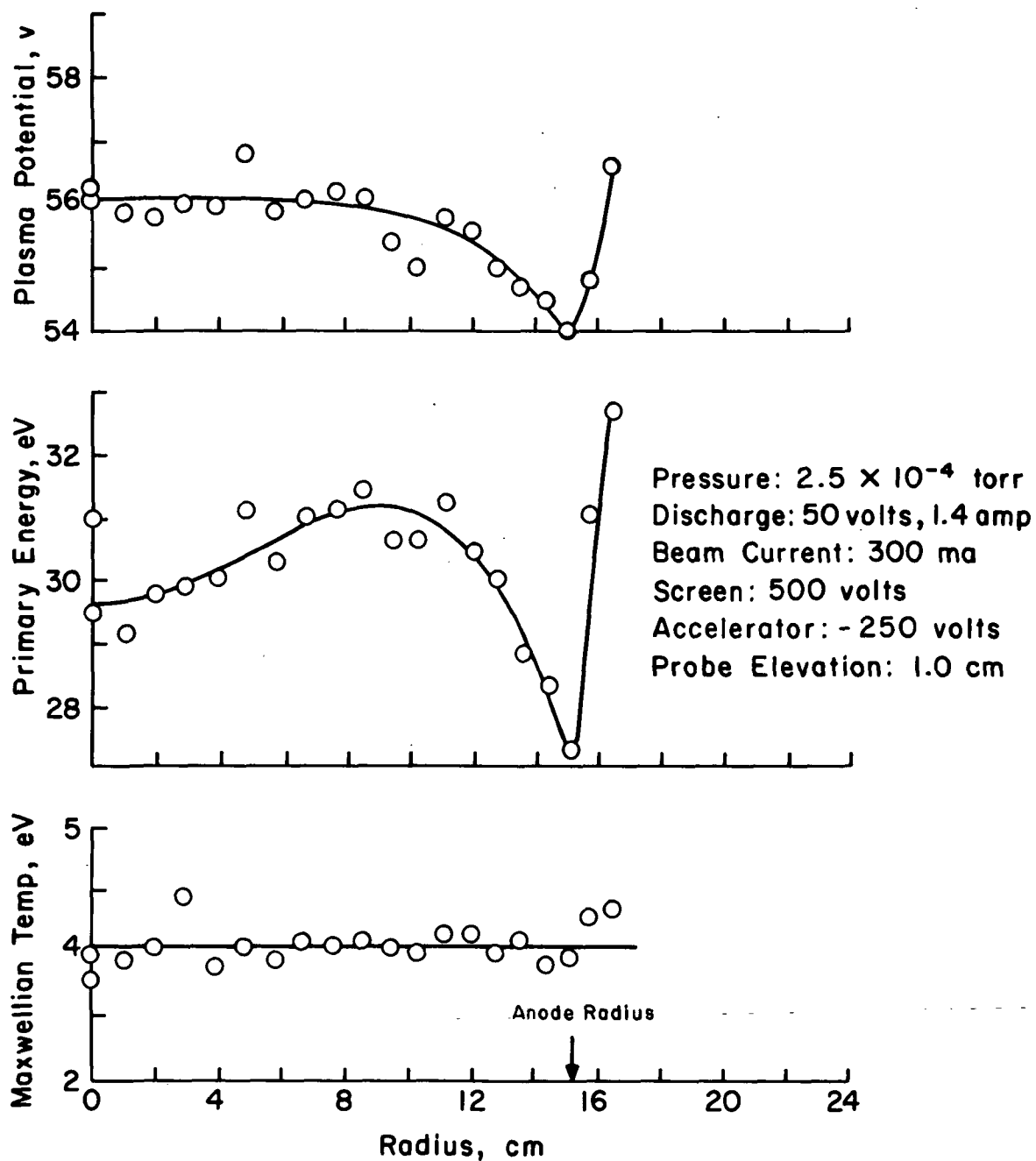


Figure 2-6. Measured plasma potential, primary energy and Maxwellian temperature as a function of radius in a plane 1 cm above the screen grid.

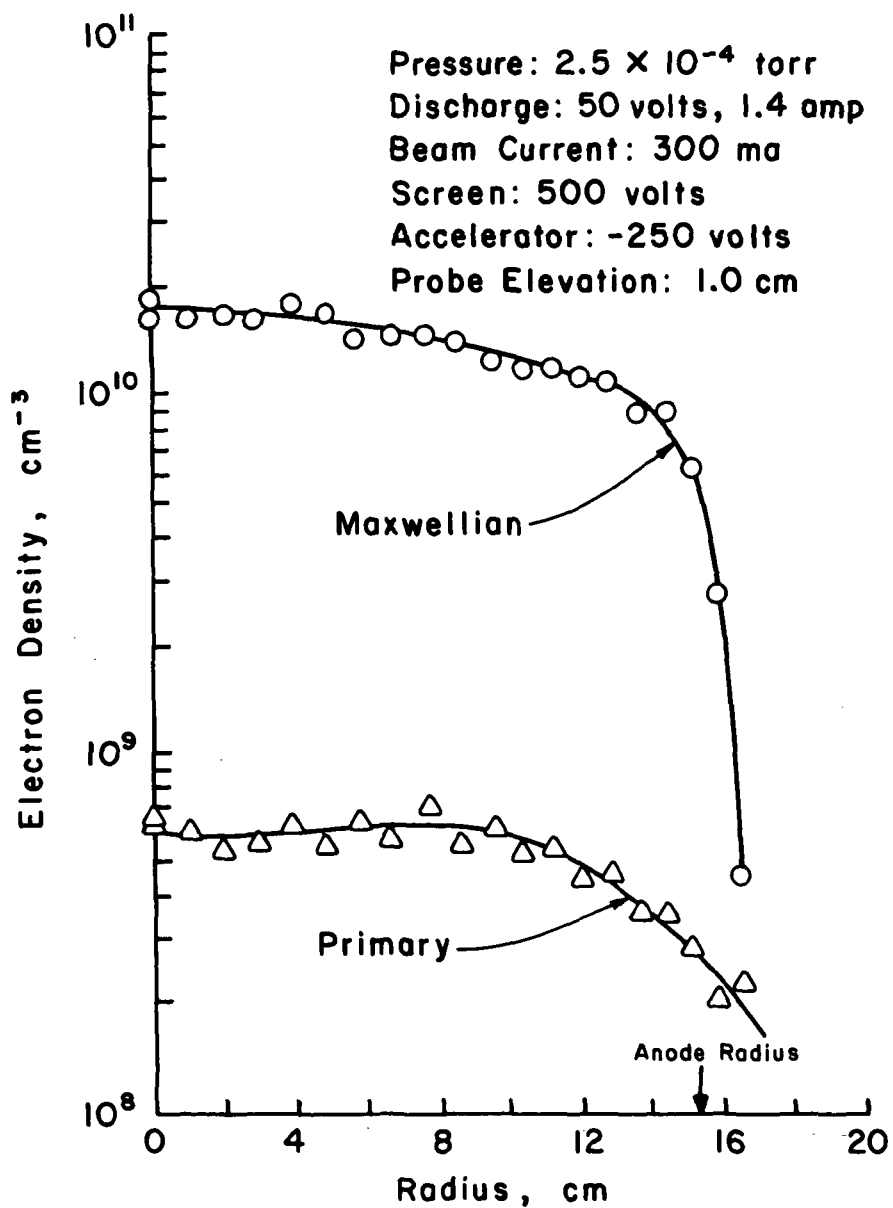


Figure 2-7. Primary and Maxwellian electron densities measured just upstream of the accelerator system as a function of radius.

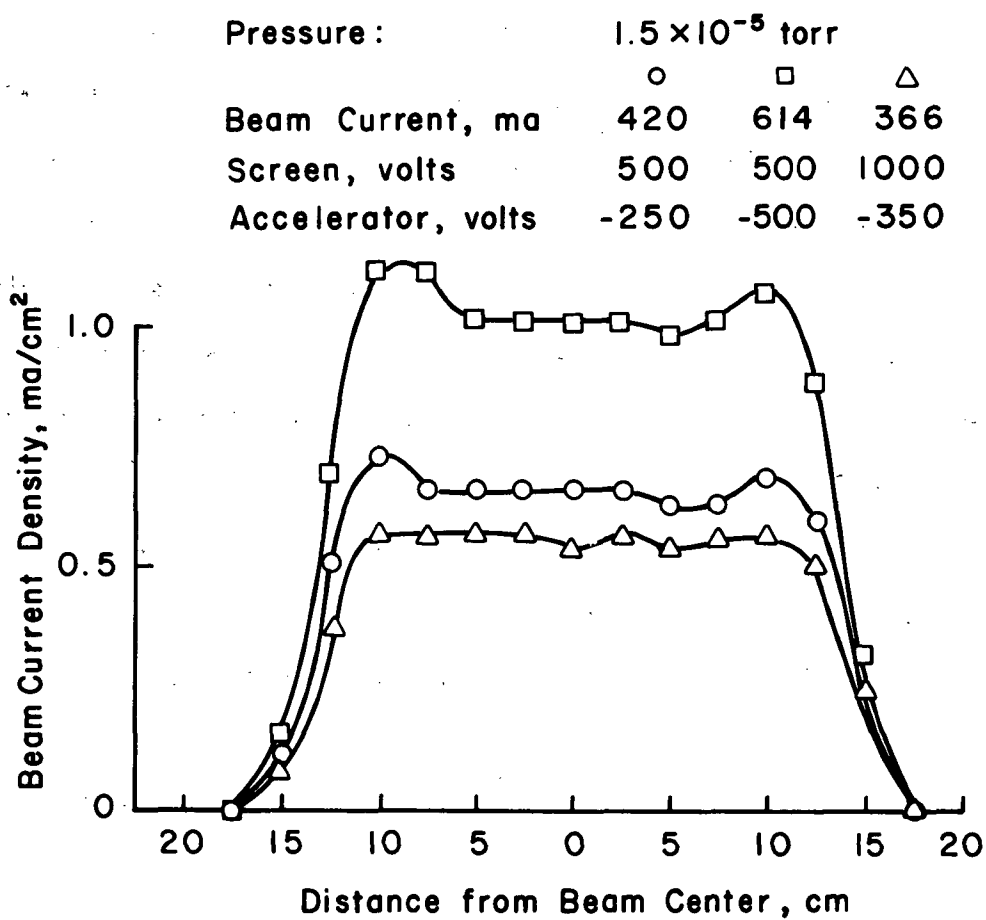


Figure 2-8. Beam current density profiles measured for the 30-cm ion source.

III. FIFTEEN CM ION SOURCE

by

Curtis M. Haynes

Source Description

A 15 cm* ion source was designed and fabricated for use in sputter etching and surface texturing applications of this grant. A uniform ion beam current density profile and high current density were of primary importance in the design of the ion source. The multipole approach was chosen to enhance the uniformity of the plasma in the discharge chamber, hence the uniformity of the extracted beam. The permanent magnet multipole design used is similar in many details to that used in an earlier 30-cm ion source.¹

A cross-section of the 15-cm discharge chamber is shown in Fig. 3-1. Alnico V magnets were used between low-carbon steel pole pieces to provide the multipole field. A thermionic cathode is supported from four insulated supports extending into the discharge chamber. Argon working gas is fed directly into the discharge chamber through four ports located in the upstream end of the chamber.

Unlike earlier multipole designs,^{1,2} anodes were located only around the cylindrical side wall of the chamber. The omission of anodes (and pole pieces) at the upstream end of the discharge chamber involved

*Ion source size has customarily been expressed as the diameter of the discharge chamber. However, an accelerator system can be masked down to any desired aperture. The '15 cm source' should more correctly be termed a '10 cm source' with the masking used herein.

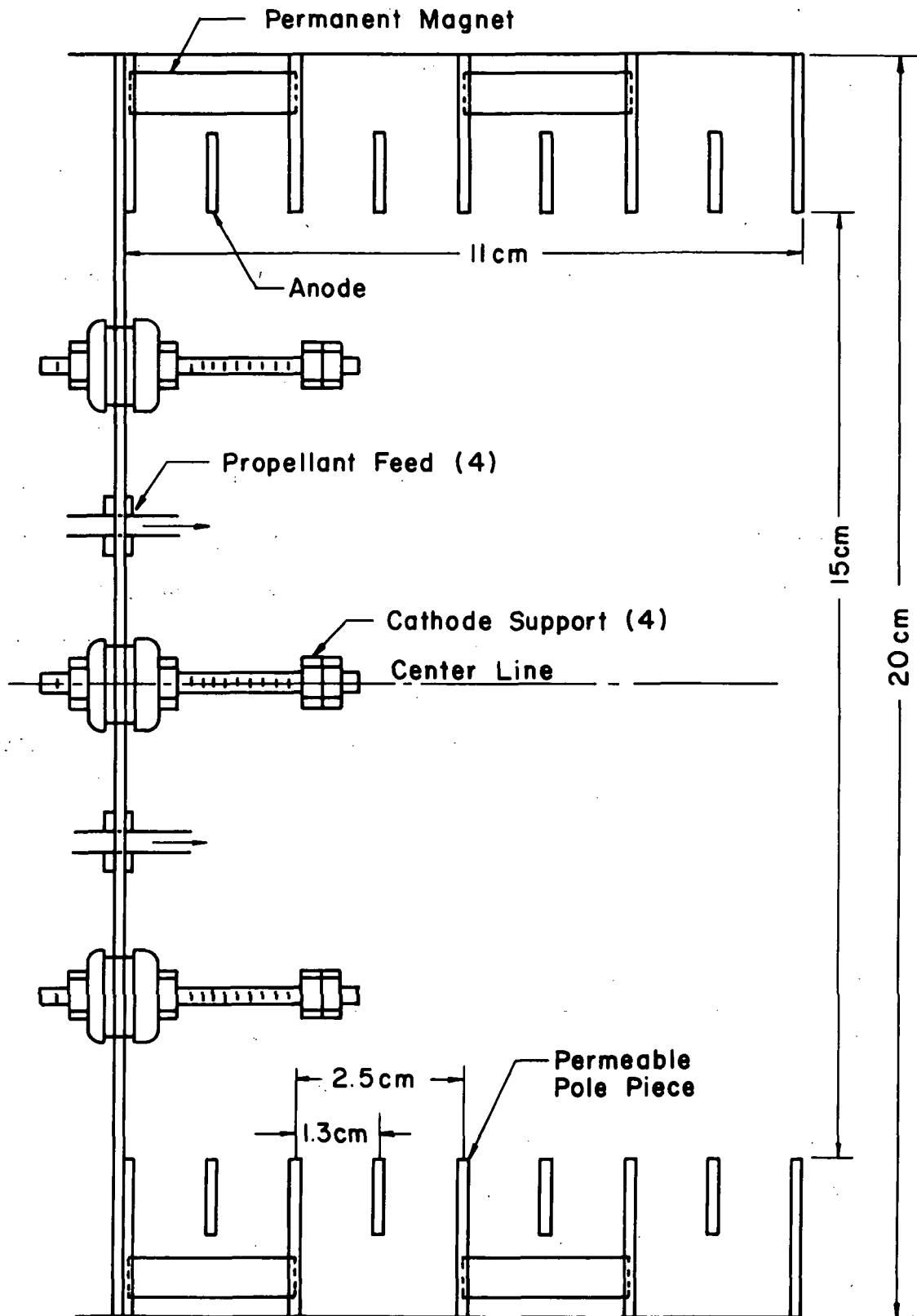


Figure 3-1. Cross section of the 15-cm discharge chamber.

a small technical risk and was done to simplify fabrication. The earlier 30-cm design showed degraded performance and more restrictive operational limits when anodes were electrically disconnected.³ In fact, the 30-cm ion source would not operate with all the pole pieces on the upstream end disconnected. A 15-cm electromagnet version of the multipole discharge chamber,² however, was not nearly as sensitive to anode changes. It was reasoned (correctly) that omission of anodes on the upstream end would not be serious for the 15-cm size. This omission also eliminated the problem of magnetic field distortion at the back and side wall corner.

The screen grid and accelerator grid were of dished molybdenum 0.4 mm thick. The grids were masked with tantalum foil to leave a 10-cm diameter extraction area. This was done to reduce the bell jar pumping capacity necessary to operate the source while still extracting the beam from the most uniform plasma region in the center of the discharge chamber. The grids had identical non-compensated aperture patterns with 1.9 mm diameter holes on 2.2 mm centers in a hexagonal array. The open area fraction of each grid was 67 percent.

Source Operation

The 15-cm ion source was operated first with argon as the working gas in a bell jar pumped with a 6 in. diffusion pump. Figure 3-2 is a plot of discharge loss as a function of total beam current over a typical range of operating pressures for this ion source. The discharge losses are higher than those measured for the 30-cm source.³ Most of this difference is probably due to the reduced 10-cm beam compared to the 15-cm chamber diameter.

Operation with tetrafluoromethane (CF_4) resulted in discharge losses ranging from 720 eV/ion to 950 eV/ion for beam currents up to 100 ma.

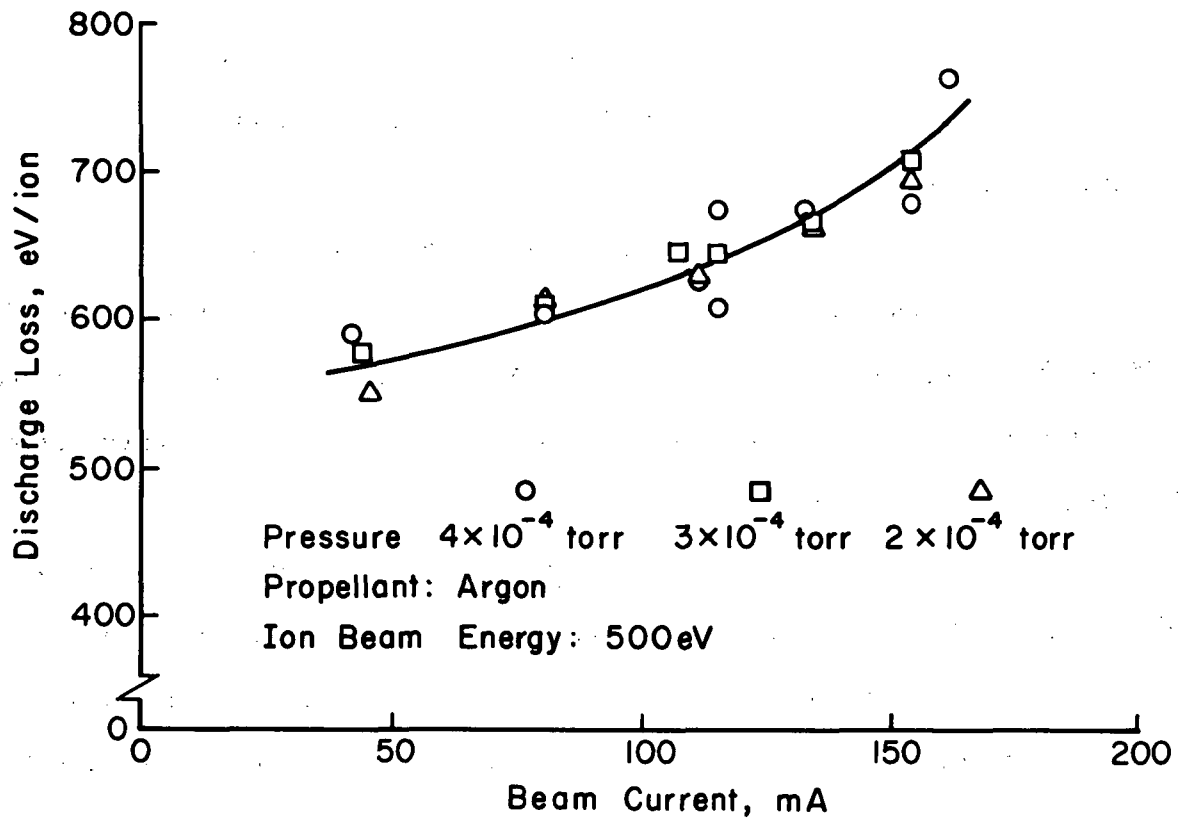


Figure 3-2. Discharge loss for the 15-cm chamber as a function of total beam current using argon as the working gas.

It was also necessary to operate at about double the indicated pressure used for argon operation. A steady degradation in ion source performance with time was noted while using CF_4 . This problem was traced to coatings on discharge chamber surfaces and corrected by cleaning.

The reduced maximum current extraction with CF_4 compared to Ar suggests that the ionized species are more massive when CF_4 is used. Assuming single ionization, preliminary scaling calculations suggest heavy ions such as CF_3^+ .

Figure 3-3 shows beam current density profiles using Ar as the working gas and taken with a moveable Faraday probe. At a distance of 5-cm from the source, the profile is uniform within ± 5 percent over the center 10 cm of beam diameter which is equal to the nominal extraction diameter of the grids.

The three profiles shown in Fig. 3-3 were all taken at the same operating conditions (discharge: 47 volts, 1.5 amps), except that at the 5 and 10 cm probe positions the pressure was 4×10^{-4} Torr and at the 15 cm probe position the pressure was 2.8×10^{-4} Torr. The total measured beam current for each probe distance was found by numerical integration to be 98.7 ma, 85.1 ma and 86.2 ma for 5, 10, and 15 cm respectively. The reduction in measured current was found to be primarily the result of charge exchange between beam ions and the background gas (see Section V).

The 15-cm source relied on previous multipole experience. It was found, after design and fabrication, to operate satisfactorily for the desired applications.

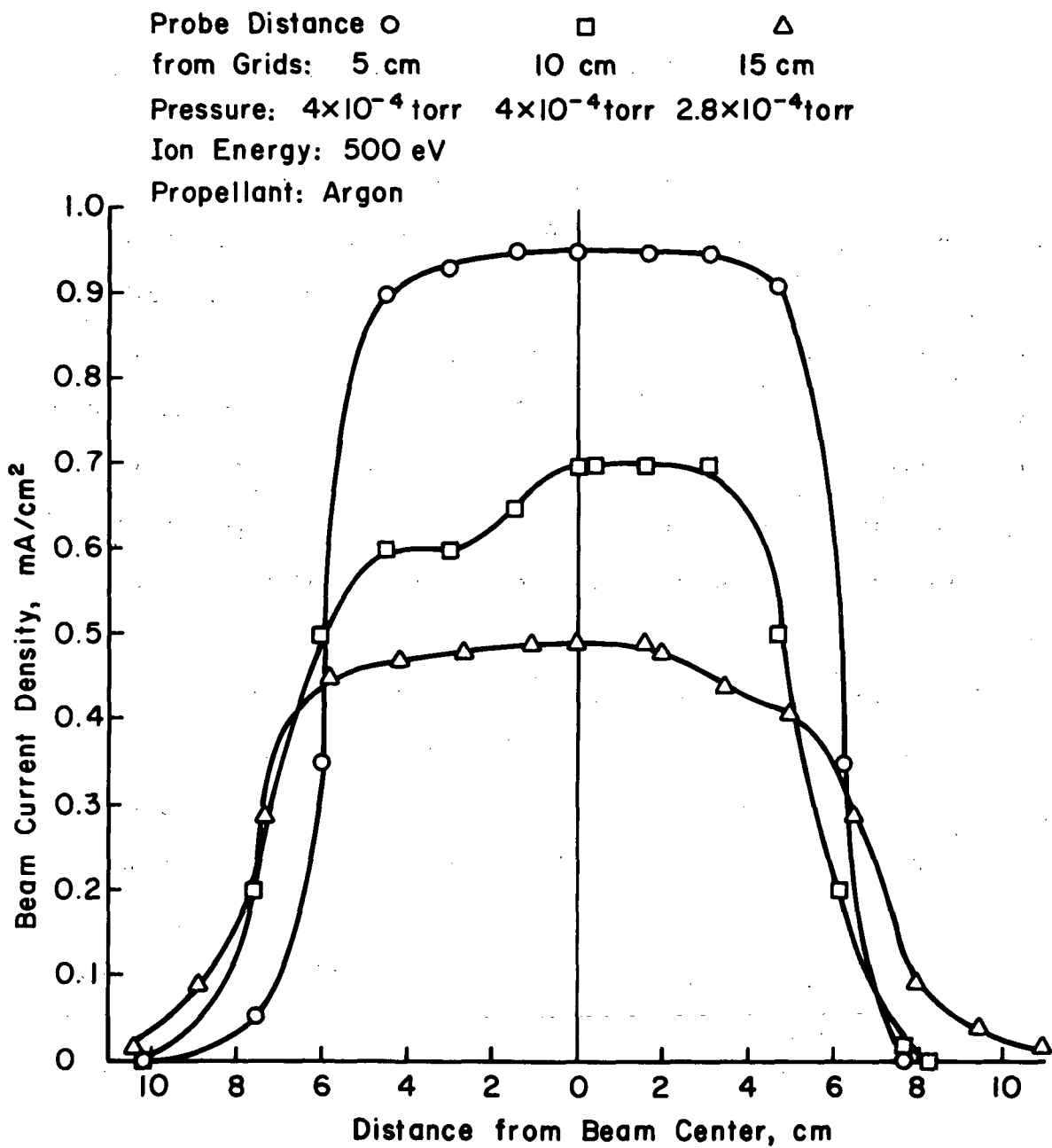


Figure 3-3. Beam current density profiles measured at three distances from the 15-cm source.

IV. SURFACE TEXTURING

by

Raymond S. Robinson and Curtis M. Haynes

A preliminary survey of surface texturing with a directed ion beam was conducted using several seed materials and a variety of surface materials. The combinations of seed and surface materials described herein were selected as rapidly revealing the maximum texturing information. Argon was the ion source working gas during most of the survey, although tetrafluoromethane (CF_4) was used for some runs to obtain a comparison with reactive etching. Because of the preliminary nature of the survey, the emphasis was on qualitative, rather than quantitative, results.

This survey, as mentioned above, is preliminary in nature. The end objective of a more complete surface texturing study would be to develop convenient and reliable quantitative procedures for obtaining a variety of textured surfaces. The potential applications of these textured surfaces would include control of secondary electron emissions, increased absorption of light, and improved biological tissue attachment (in medical implants).

Apparatus and Procedure

The runs with Ar as the working gas were conducted with a 30 cm ion source, while those with CF_4 were conducted with a 15 cm ion source. Both ion sources used dished molybdenum accelerator systems with screen and accelerator grid potentials of +500 and -250 V. Ignoring a small fraction of doubly ionized atoms or molecules, the energy of all beam

ions was therefore 500 eV. Both ion sources were also operated in 45-cm diameter bell jars. Ion-beam current densities were measured with Faraday probes that were 7 and 10 cm from the grids for the 15 and 30 cm ion sources, respectively. The ion beams were neutralized with electrons emitted by heated refractory filaments that were located in the ion beam 2-3 cm from the accelerator grid. The indicated pressures in the bell jars were 3×10^{-4} torr for Ar and approximately 9×10^{-4} torr for CF_4 .

The surface materials included in the survey were Si, Mo, Ta, stainless steel (S-S), Al, C (graphite form), Cu, and brass. The samples sputtered were 1.3 cm square sections sheared from larger sheets, except for Si which was available in irregular pieces. Before sputtering, each sample was cleaned in trichloroethane, acetone, and then water. The Cu was subjected to an additional initial cleaning step using a 50 percent solution of nitric acid. Shields, that also served as sources of seed material, were made of S-S, Mo, Ta, Cu, and C. The shields were located so that only half of each sample was exposed to the ion beam. The samples were located 30 cm from the 30 cm ion source and 10 cm from the 15 cm ion source. A shutter was used to precisely control exposure to the ion beam.

A schematic of the stainless steel sample holders used in this investigation is shown in Fig. 4-1. Sample holders were fabricated with the tilt angle θ varied from 0 to 50° in 5° steps. Separate holders were used to set the angle of beam incidence on each sample, although most sputtering was at normal incidence. The edge of the shield was a source of seed material for the sample, with the intensity of seeding varying with distance from the edge of the shield. The covered half of the sample served as a control surface. The sputtered surfaces and the

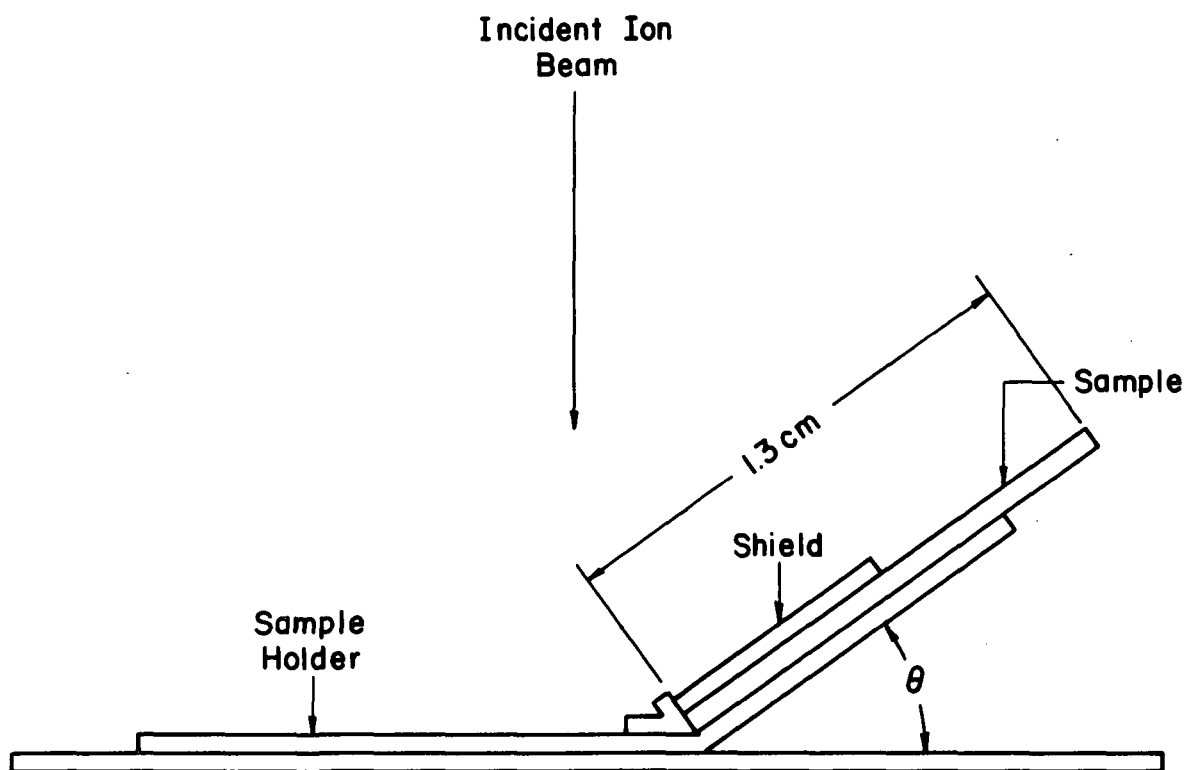


Figure 4-1. Sample holder used for texturing materials.

nonsputtered control surfaces were examined using a scanning electron microscope. The micrographs were taken at various tilt angles to enhance the visibility of different types of surface morphology. All sputtering was done without heat sinks at an estimated sample temperature of 200-300°C.

Table 4-1 shows estimated etch depths for the materials tested with Ar as the working gas. These etch depths were estimated using tabulated values for the sputter yields of the materials along with atomic weight and density data. Reported stainless steel sputtering rates differ widely. As a first approximation for stainless steel, data for Fe have been included in Table 4-1.

TABLE 4-1. Estimated Sample Etch Depths for 500 eV Ar Ion Beam.

<u>Material</u>	<u>Etch depth for etch dose of 200 mA-min/cm²</u>	<u>Etch depth for etch dose of 240 mA-min/cm²</u>
C	0.8 μ	1.0 μ
Al	13.	16.
Si	6.4	7.7
Fe	8.6	10.
Cu	16.	20.
Mo	8.4	10.
Ta	7.7	9.3

A similar estimate of etch depth cannot be made with CF₄ as the working gas because the sputtering yield of the ions produced from CF₄ is not known. The etch doses for the various tests are given in mA-min/cm², as shown in Table 4-1. The tests were conducted at 1 mA/cm² for the Ar tests and 0.2 - 0.8 mA/cm² for CF₄. Rate dependent effects would not normally be expected, so that the integrated doses in

mA-min/cm^2 were felt to be more meaningful than the various current densities and times used to obtain the doses. The typical dose in terms of ions/cm^2 was a large number in this study. For example, a mean flux of 1 mA/cm^2 for one hour would represent a dose of 60 mA-min/cm^2 in the units used herein. Translated into ions/cm^2 , this is 2.25×10^{19} .

This means that each square angstrom or, roughly, each atomic site is bombarded by more than two thousand ions. From the scale of etch patterns obtained and estimates of the material removed, the etch patterns should be near steady-state morphology and therefore not change significantly for larger etch doses.

Results

Silicon

A single-crystal Si wafer was sputtered at normal incidence with an etch dose of 200 mA-min/cm^2 using Ar as the working gas and with an etch dose of 20 mA-min/cm^2 using CF_4 as the working gas. Figure 4-2 is representative of the Si surfaces after sputtering with both Ar and CF_4 working gases. This micrograph shows the transition between the sputtered region on the left and the nonsputtered control surface on the right. No topographical differences could be detected between the sputtered and control surfaces, even at higher magnifications. Seed (shield) materials included S-S, C, Cu, Ta, and Mo. The seeding close to the shield edge was sufficient to impart the characteristic color for the seed material to the sputtered Si. It is therefore unlikely that a further increase in seeding intensity alone would induce texturing. The illumination in the micrographs depends on secondary electron emission rather than visual color; but, in all cases the sputtered areas in the micrographs were darker than the control areas near the transition between the two.

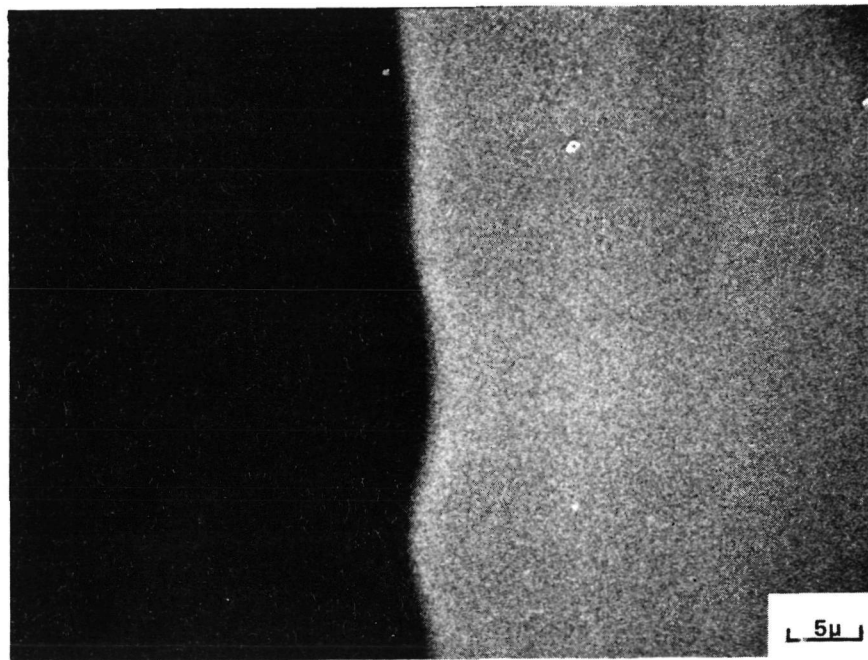


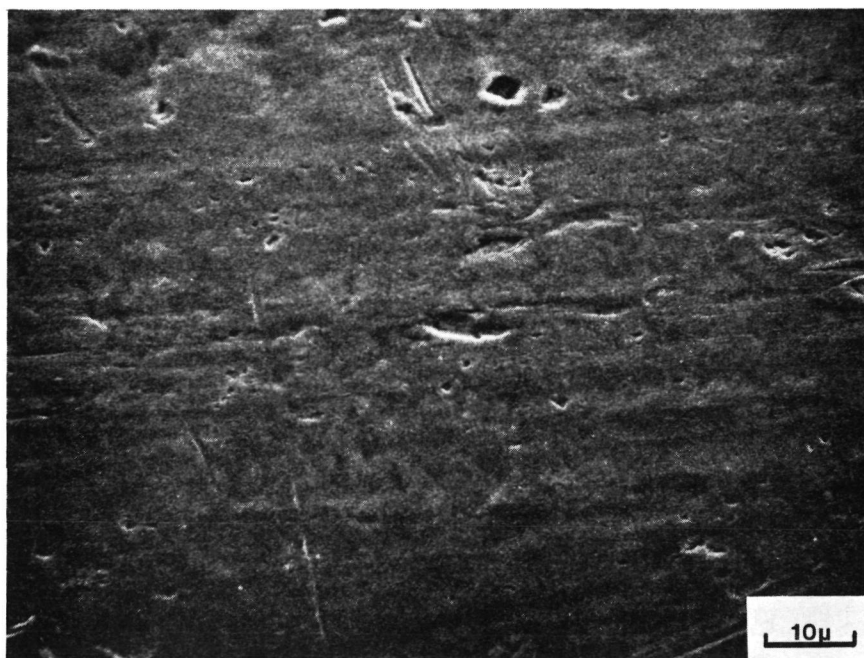
Figure 4-2. Transition between unspattered (right) and spattered silicon, normal incidence. Typical of sputtering using both Ar and CF_4 as the working gases.

Molybdenum

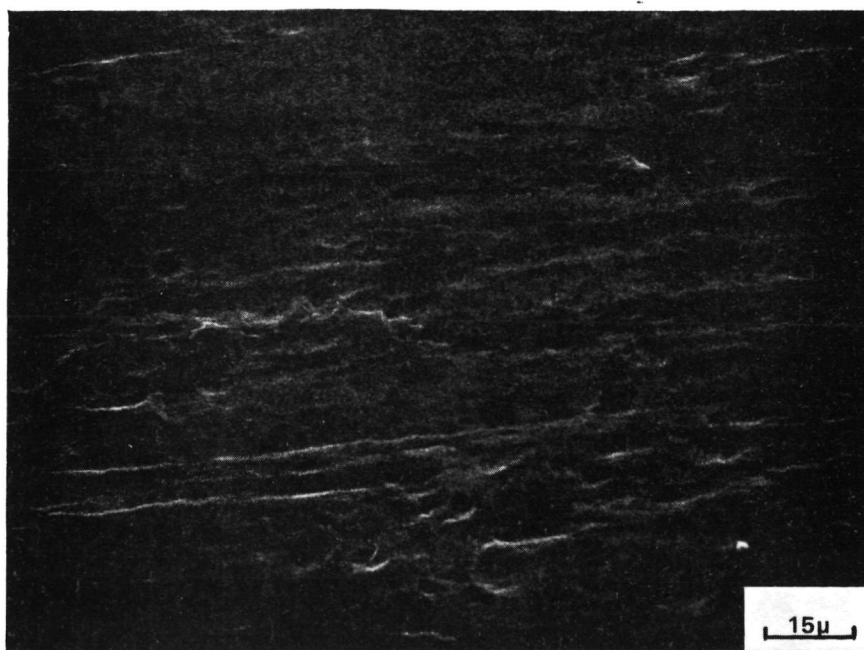
Samples of Mo were sputtered with an etch dose of 200 mA-min/cm^2 using Ar as the working gas and an etch dose of 20 mA-min/cm^2 using CF_4 as the working gas. Figure 4-3(a) shows a micrograph of the nonsputtered control surface.

A sample of sputtered Mo is shown in Fig. 4-3(b). Only a slight change in morphology can be detected relative to the Mo control surface. The sputtered Mo surface has shallow pits that were not evident on the control surface. The appearance with a S-S shield was similar to that shown in Fig. 4-3(b).

Figure 4-3(c) is a micrograph of a Mo surface after sputtering with CF_4 as the working gas. No significant change is apparent relative to the control surface in Fig. 4-3(a). The small pieces of material that appear to be resting upon the surface in Fig. 4-3(c) are perhaps contamination received after the sputtering.

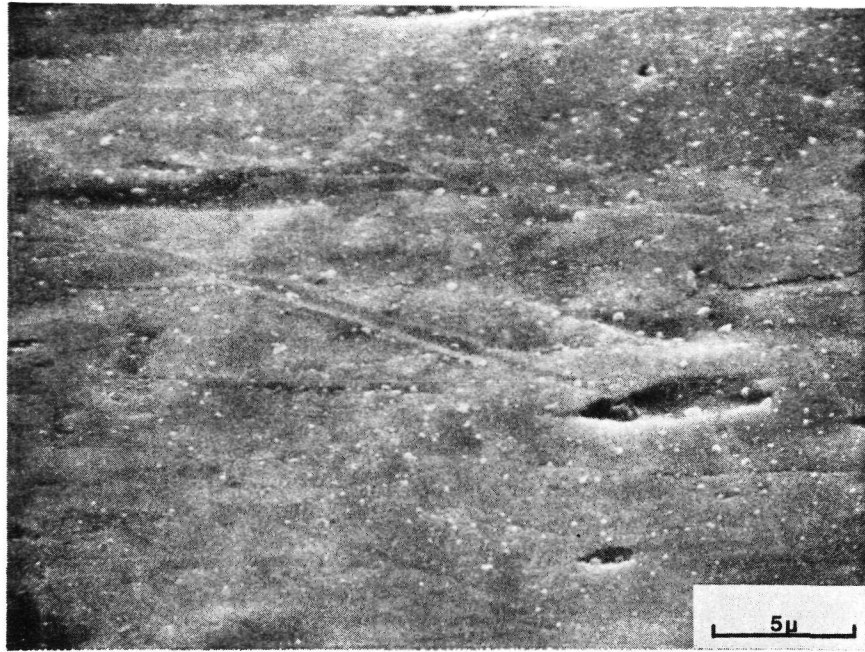


(a) Non-sputtered, control surface.



(b) Sputtered, Ar used as the working gas, with etch dose of 200 mA-min/cm^2 .

Figure 4-3 Molybdenum samples. Sputtering was at normal incidence.



(c) Sputtered, CF_4 used as the working gas. Stainless steel shield, with etch dose of 20 mA-min/cm^2 .

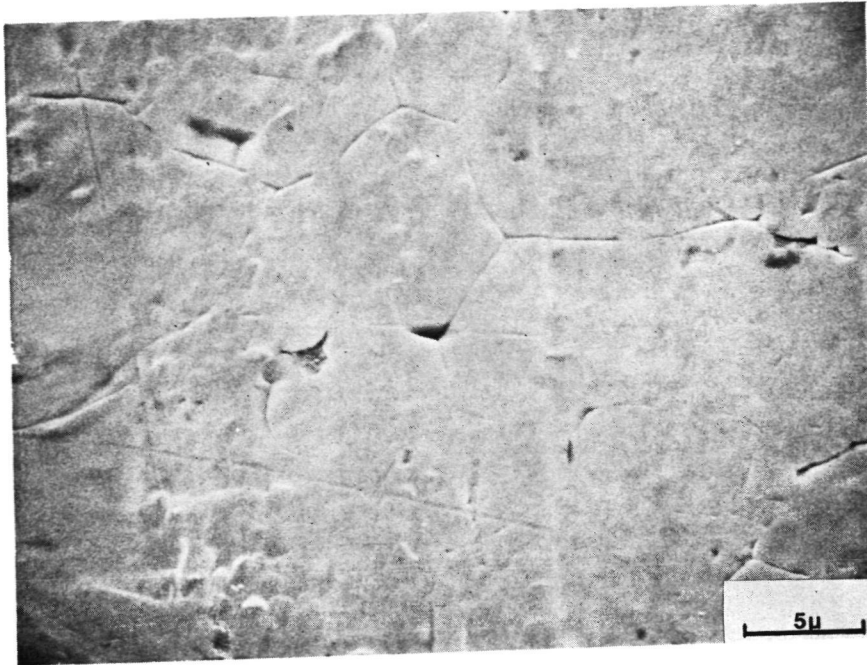
Tantalum

Samples of Ta were sputtered with an etch dose of 200 mA-min/cm^2 using Ar as the working gas and an etch dose of 20 mA-min/cm^2 using CF_4 as the working gas. Both S-S and Mo were used as shields. The micrograph of the nonsputtered control surface is shown in Fig. 4-4(a). Rough polygons are evident in this micrograph, and are believed to be crystal grain boundaries. The visual appearance of this nonsputtered surface was shiny.

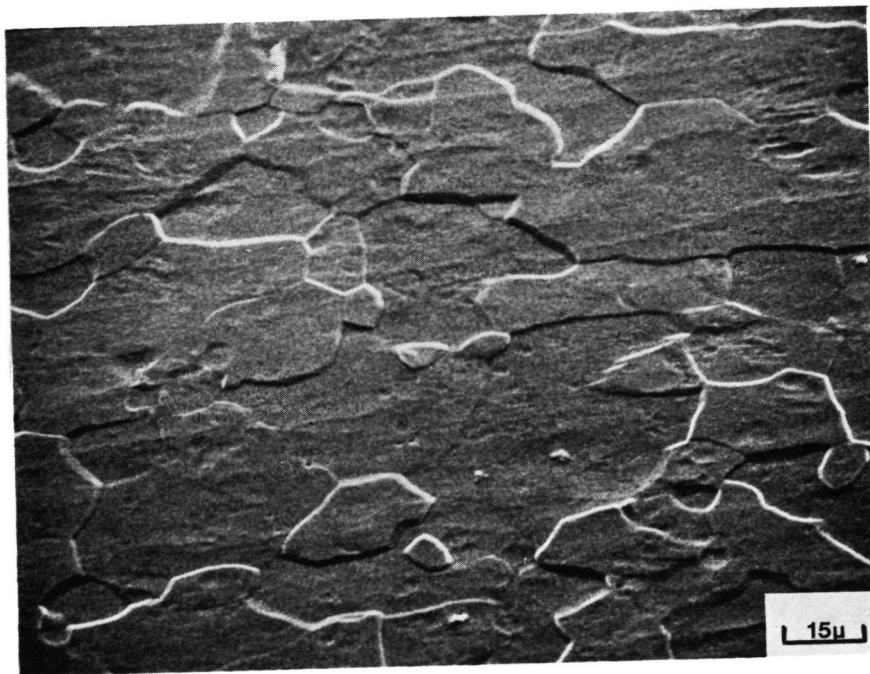
The Ta surface after sputtering with Ar is shown in Fig. 4-4(b). The shield material was Mo for Fig. 4-4(b), but the surface appearance was similar with a S-S shield. The polygon boundaries are similar to those shown in Fig. 4-4(a), but the various crystal grains appear to have etched at different rates. These different etching rates are believed due to the variation of sputtering yield with crystal orientation.

Visually, the sputtered Ta surface was gray and dull relative to the shiny control surface. A dark gray line about 0.01 mm wide was visible at the shield edge. A less intense region about 1 mm wide extended out from the dark gray line.

The Ta surface after sputtering with CF_4 is shown in Fig. 4-4(c). The polygon crystal boundaries are again visible, but the different crystal boundaries do not exhibit different etch depths. Darker regions are also visible in Fig. 4-4(c), but the cause of these darker regions is not known.

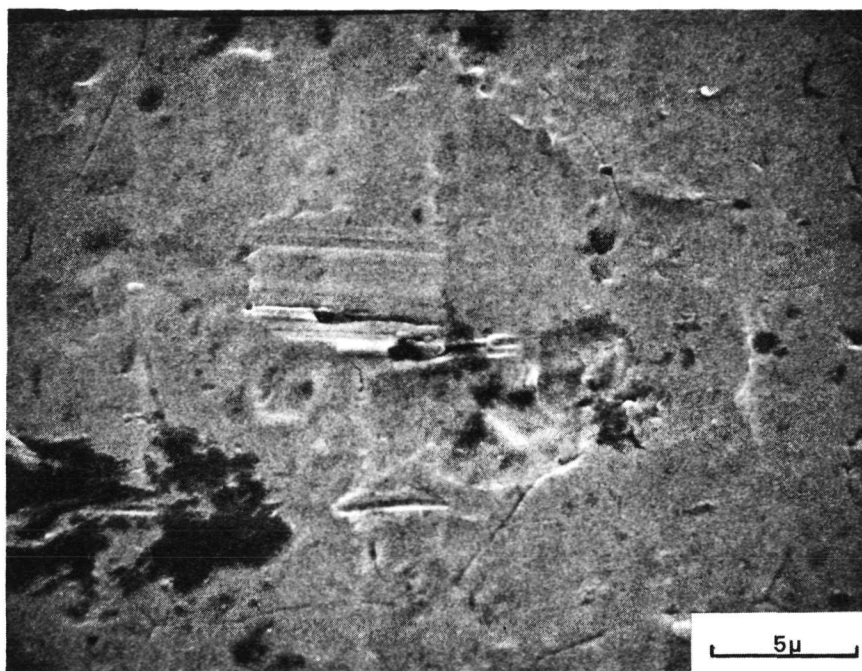


(a) Nonsputtered control surface.



(b) Sputtered, Ar used as the working gas. ^{90}Mo Molybdenum shield, with etch dose of 200 mA-min/cm². (Appearance was similar with stainless-steel shield.)

Figure 4-4 Tantalum samples. Sputtering was at normal incidence.



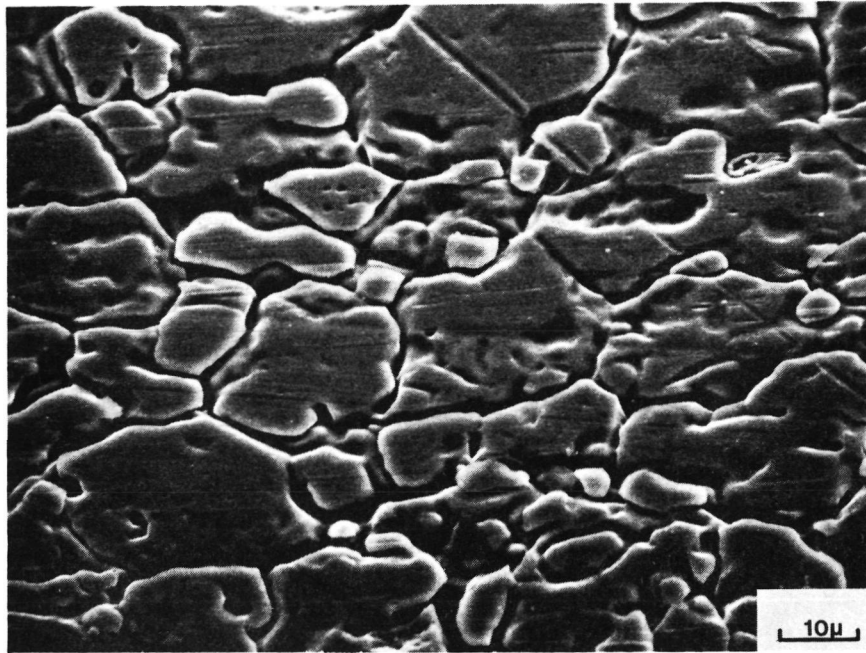
(c) Sputtered, CF_4 used as the working gas. Stainless-steel shield, with etch dose of 20 mA-min/cm^2 .

Stainless Steel

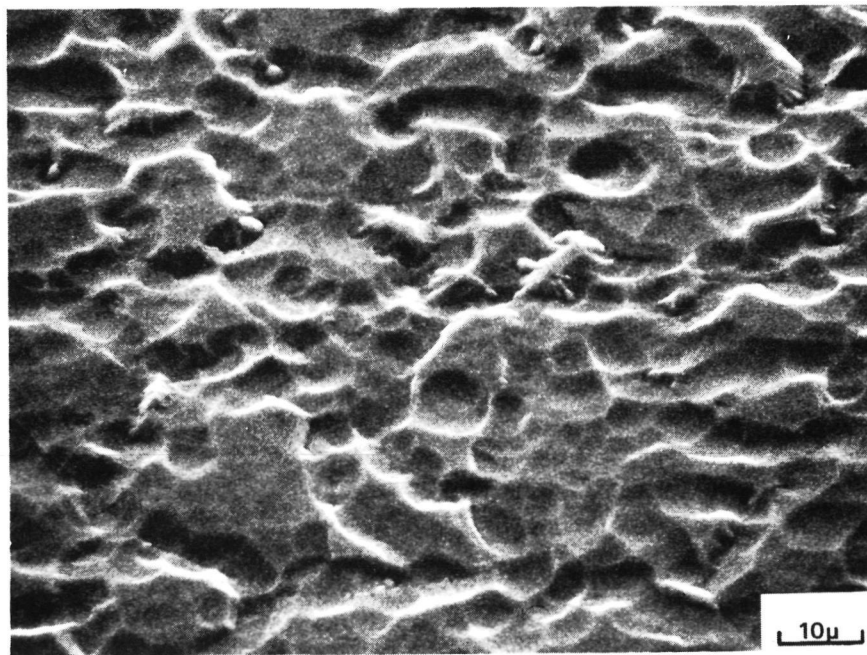
Samples of S-S were sputtered with etch doses of 200 and 240 mA-min/cm² using Ar as the working gas and with an etch dose of 24 mA-min/cm² using CF₄ as the working gas. The latter etch dose was obtained with 0.8 mA/cm² for 30 min. A CF₄ etch for 100 min. at 0.2 mA/cm² (etch dose, 20 mA-min/cm²) was also used, but with no observable difference from the CF₄ etch dose given above. Both S-S and Mo were used for shields. The S-S used for both samples and shields was type 304 annealed sheet, 0.5 mm thick. The micrograph of the nonsputtered control surface is shown in Fig. 4-5(a). Note the distinctive pattern of irregular forms surrounded by narrow channels. This appearance was typical of all S-S control surfaces.

The S-S surface after sputtering with Ar is shown in Fig. 4-5(b). The pattern shown is one of numerous overlapping small depressions. No change in this pattern was observed near either the S-S or Mo shields.

The S-S surface after sputtering with CF₄ is shown in Figs. 4-5(c) and (d). The pattern in Fig. 4-5(c) has a fairly uniform fibrous or filamentary appearance, presumably due to the selective reactive etching of certain elemental constituents of the S-S. At the higher magnification shown in Fig. 4-5(d), the fibrous pattern is seen to have a structural scale or size similar to that shown on the control sample. This scale similarity implies that the pattern in Fig. 4-5(a) is associated with nonuniform distributions of constituent elements, and that the selective etching with CF₄ results in structures of similar scale for the etched pattern of Fig. 4-5(d).

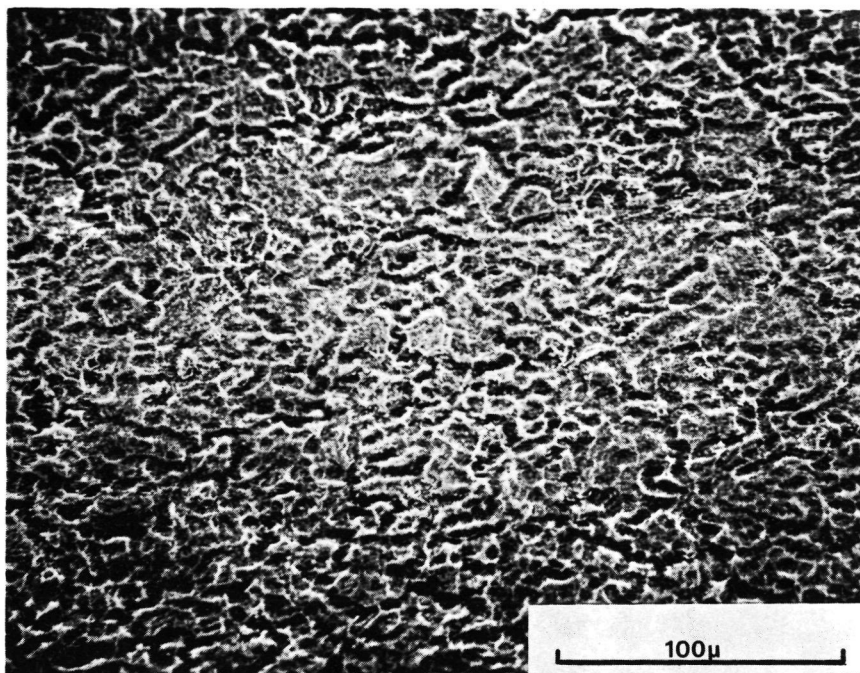


(a) Nonsputtered control surface.

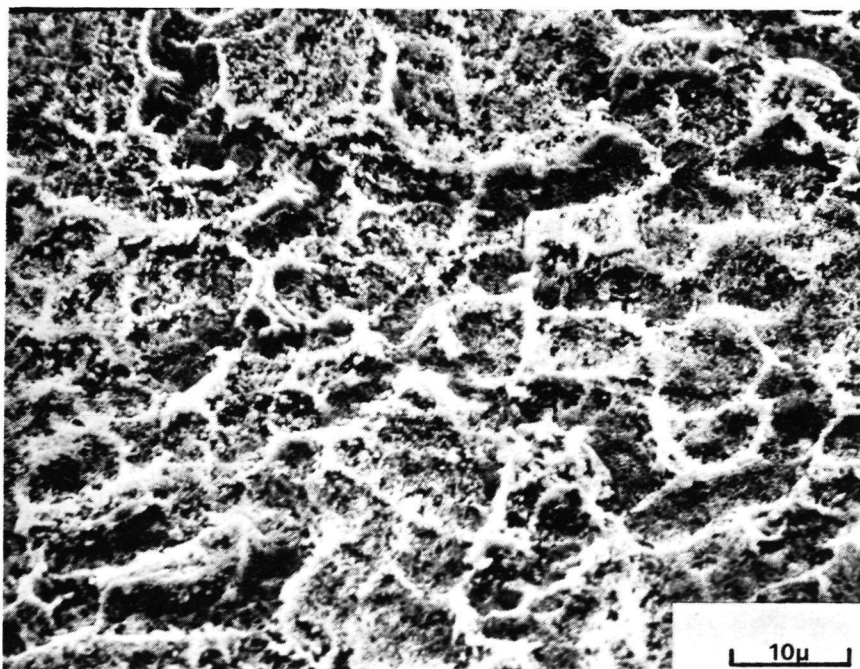


(b) Sputtered, Ar used as the working gas. Stainless-steel shield, with etch dose of 240 mA-min/cm^2 . (Appearance was similar with molybdenum shield.)

Figure 4-5 Stainless-steel samples. Sputtering was at normal incidence.



(c) Sputtered, CF_4 used as the working gas. Stainless steel shield, with etch dose of 24 mA-min/cm^2 .



(d) Sputtered, CF_4 used as the working gas. Stainless steel shield, with etch dose of 24 mA-min/cm^2 .

Aluminum

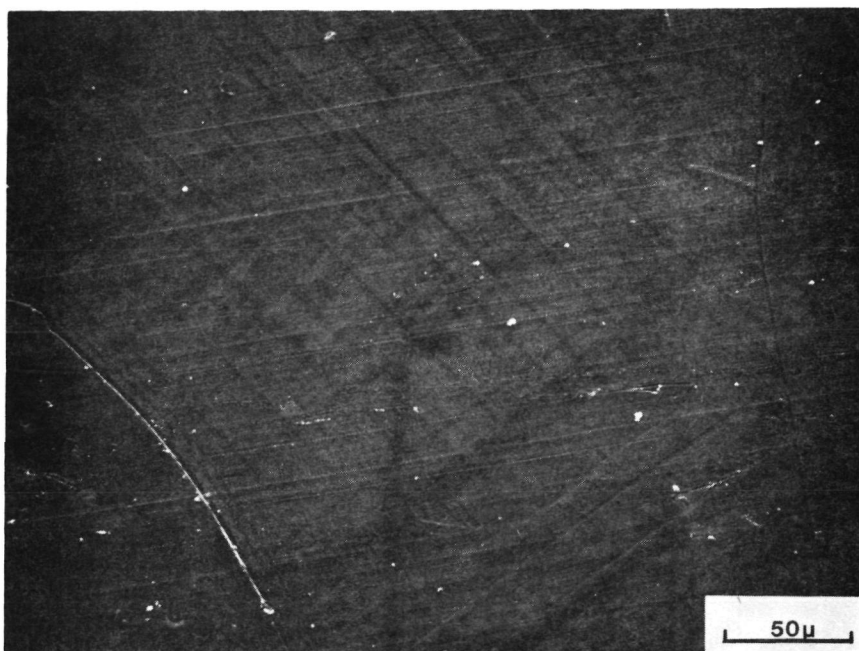
Samples of Al were sputtered with etch doses of 200 and 240 mA-min/cm² using Ar as the working gas and with an etch dose of 20 mA-min/cm² using CF₄ as the working gas. Both S-S and Mo were used as shields. The micrograph of the nonsputtered control surface is shown in Fig. 4-6(a). Except for fabrication marks and a few scratches, the control surface is uniform and smooth.

The Al surface after sputtering with Ar is shown in Fig. 4-6(b). A pattern of rough polygons, believed to be crystal grain boundaries, is clearly evident. At the increasing magnifications of Figs. 4-6(c) and (d) the surfaces within the polygons are seen to have a nearly repetitive etch pattern. In Fig. 4-6(d) the pattern consists of holes about 1 micron in diameter. Fig. 4-6(c) shows the transition from nonsputtered to sputtered regions close to the shield edge, while Fig. 4-6(d) is more representative of the surface farther from the S-S shield edge. A different pattern farther from the Mo shield is shown in Fig. 4-6(e). Although both Fig. 4-6(d) and Fig. 4-6(e) are far enough from the shield so that seeding was small, the difference in shield material (S-S and Mo) is the only obvious cause for the difference in surface texture.

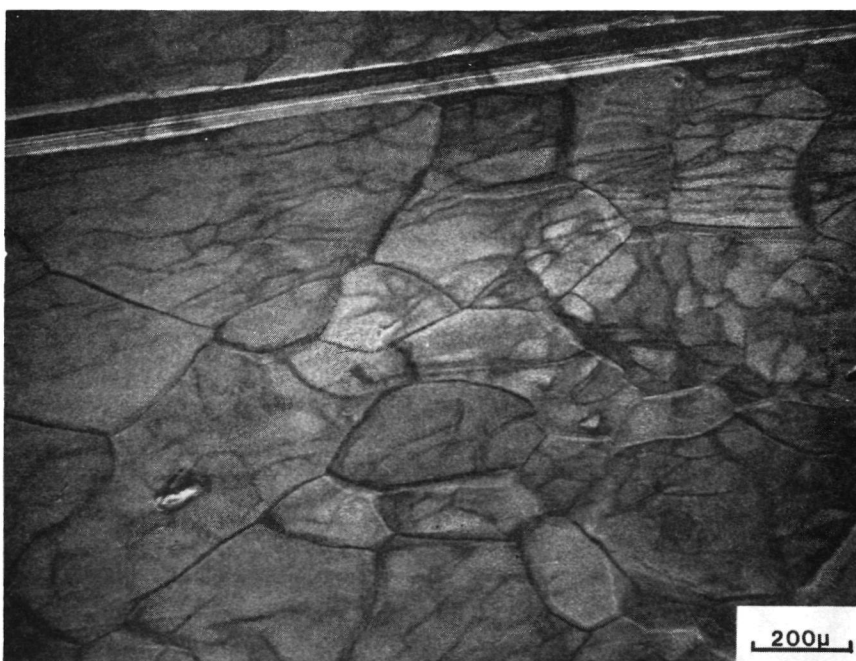
The Al surface after sputtering with CF₄ is shown in Fig. 4-6(f). No obvious difference from the Ar sputtered surface is apparent at this low magnification (see Fig. 4-6(b)). At higher magnifications, though, differences become readily apparent. The boundary between regions A and B (see Fig. 4-6(f) for region definitions) is shown in Fig. 4-6(g). Two different regular patterns are visible, with the obvious interpretation that the two patterns are associated with the different crystal orientations in regions A and B. A further enlargement of region A

is shown in Fig. 4-6(h), while a similar enlargement of region B is shown in Fig. 4-6(i). A third crystal orientation is indicated by the enlargement of region C in Fig. 4-6(j).

From the surface morphology etched with Ar (Figs. 4-6(d) and (e)) it appears that the seed material can make a significant difference in the pattern obtained. In contrast, the patterns with CF_4 at similar magnifications (Figs. 4-6(h) through (j)) appear to be due to Al crystal orientation, hence, are probably independent of seeding.

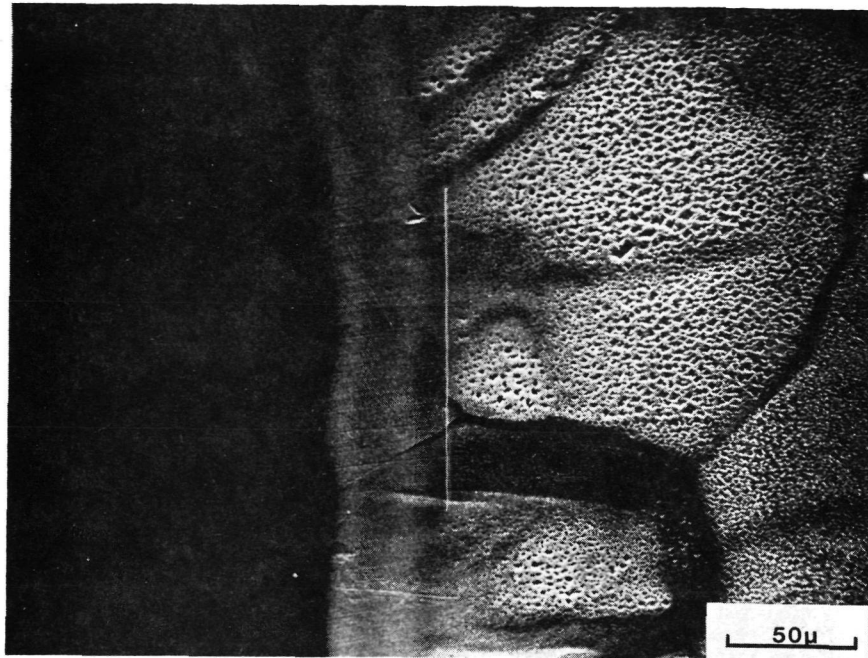


(a) Nonsputtered control surface.

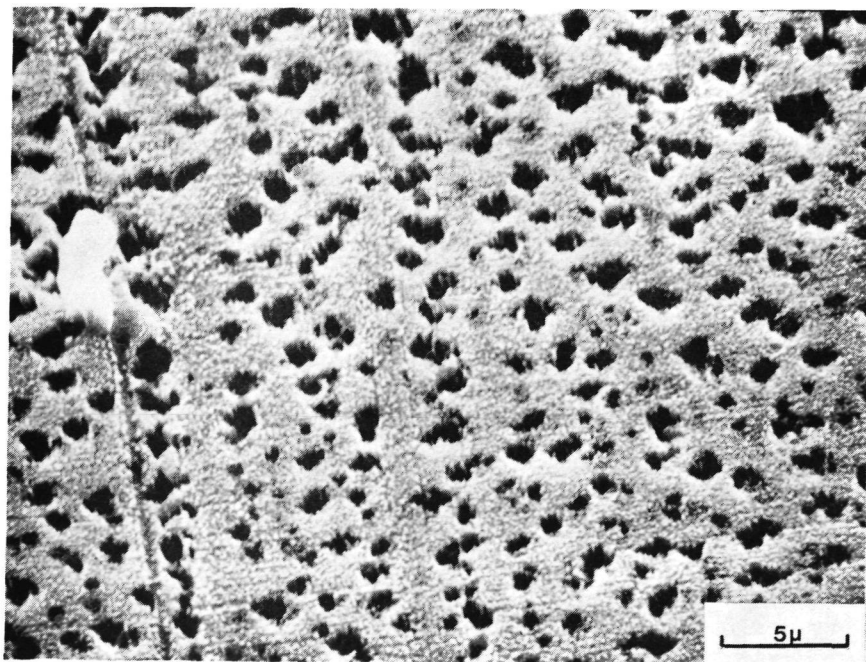


(b) Sputtered, Ar used as the working gas. Stainless steel shield, with etch dose of 240 mA-min/cm^2 .

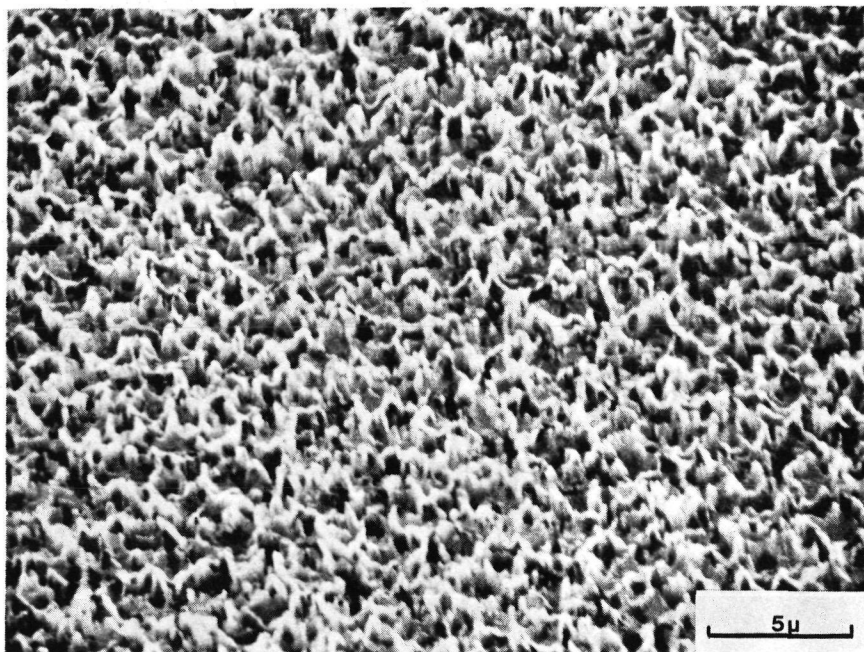
Figure 4-6 Aluminum samples. Sputtering was at normal incidence.



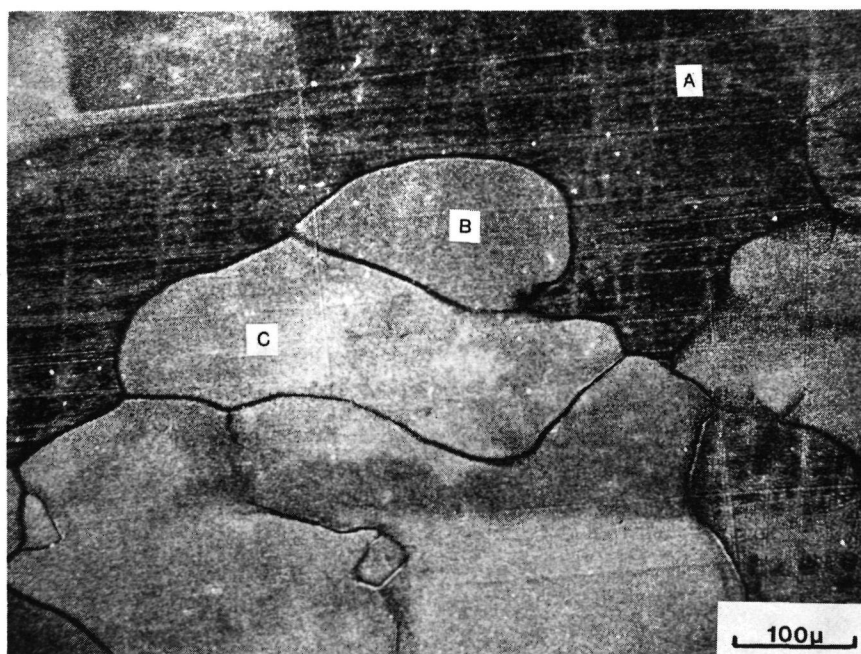
(c) Sputtered, Ar used as the working gas. Stainless steel, with etch dose of 240 mA-min/cm^2 .



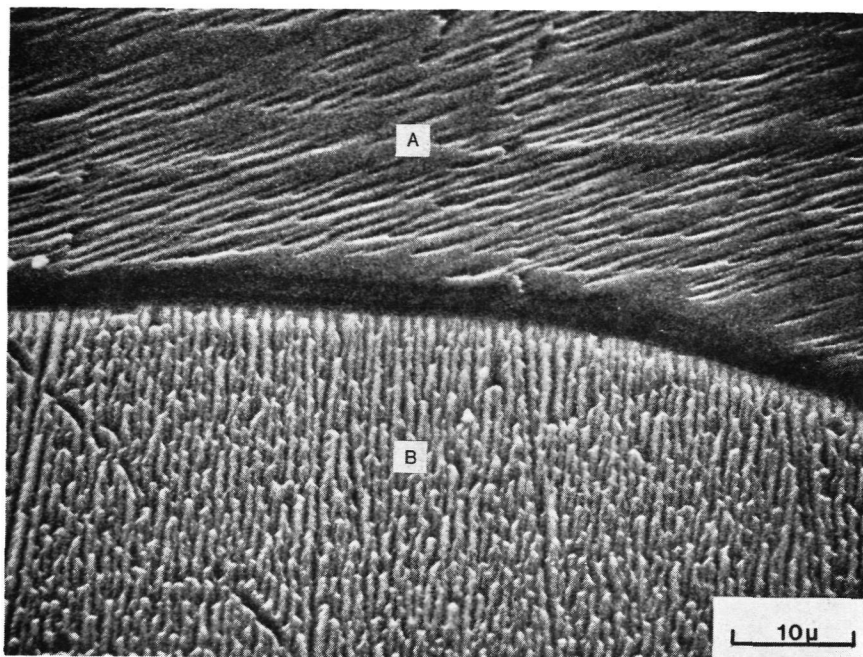
(d) Sputtered, Ar used as the working gas. Stainless steel shield, with etch dose of 240 mA-min/cm^2 .



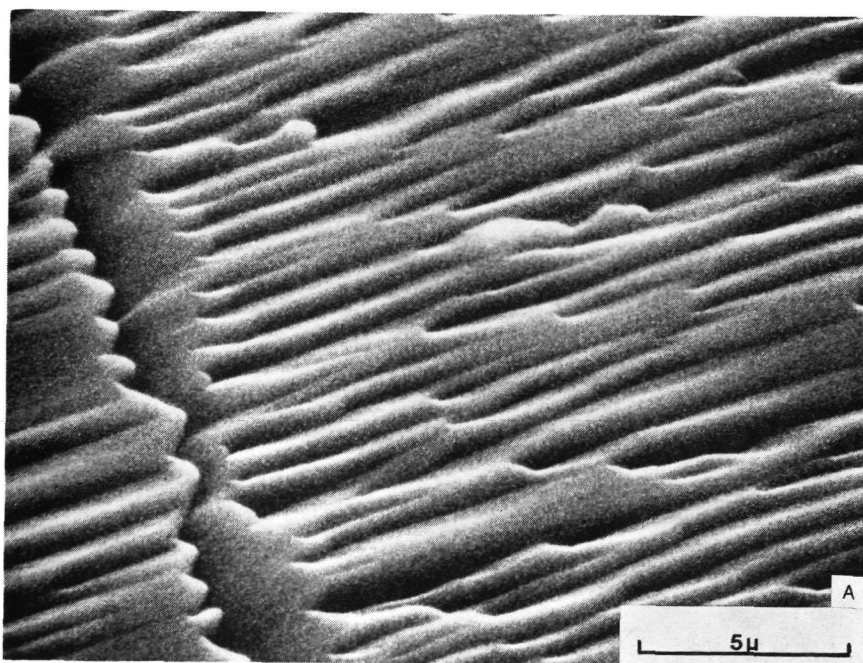
(e) Sputtered, Ar used as the working gas. Molybdenum shield, with etch dose of 200 mA-min/cm².



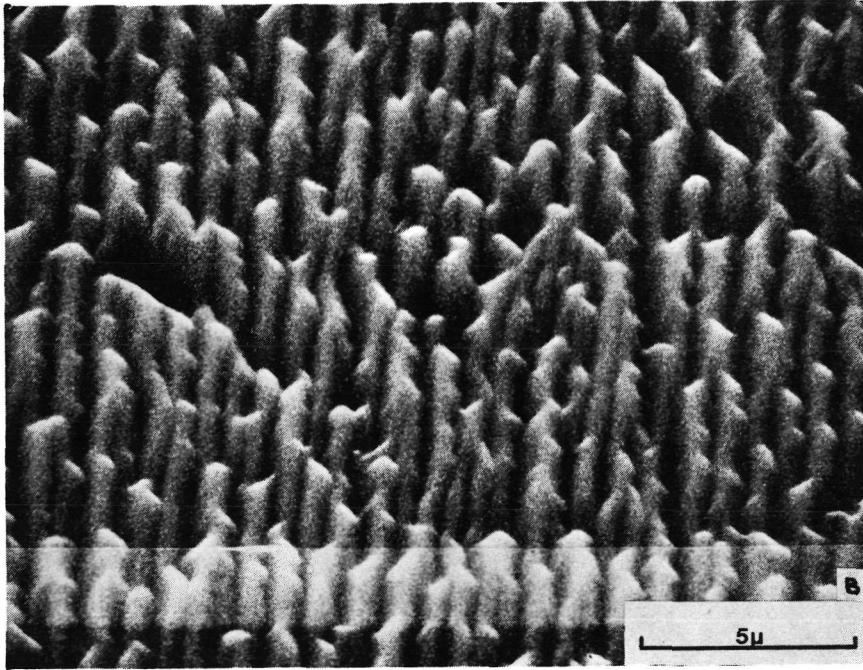
(f) Sputtered, CF₄ used as the working gas. Stainless steel shield, with etch dose of 20 mA-min/cm².



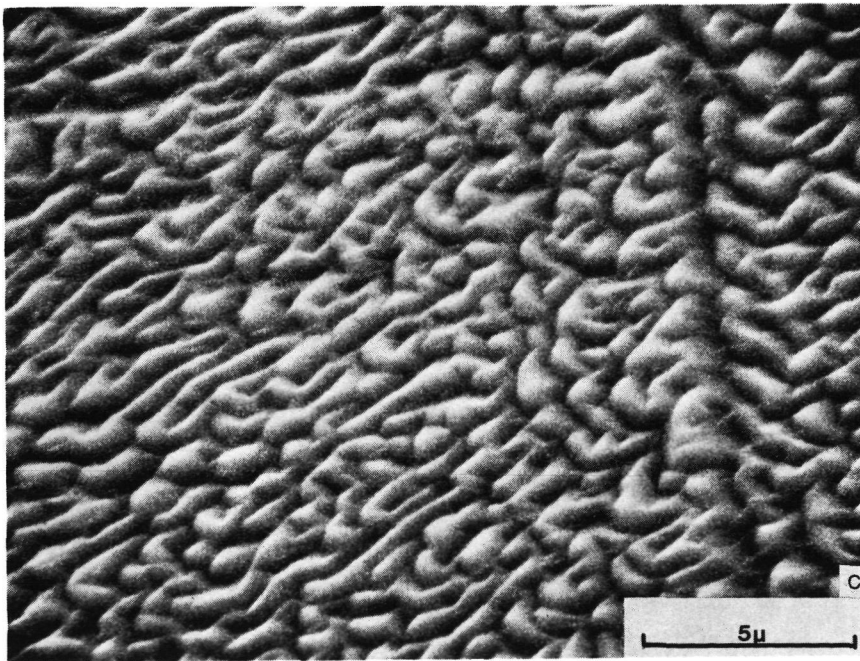
(g) Sputtered, CF_4 used as the working gas. Stainless steel shield, with etch dose of 20 mA-min/cm^2 .



(h) Sputtered, CF_4 used as the working gas. Stainless steel shield, with etch dose of 20 mA-min/cm^2 .



- (i) Sputtered, CF_4 used as the working gas. Stainless steel shield,⁴ with etch dose of 20 mA-min/cm^2 .



- (j) Sputtered, CF_4 used as the working gas. Stainless steel shield, with etch dose of 20 mA-min/cm^2 .

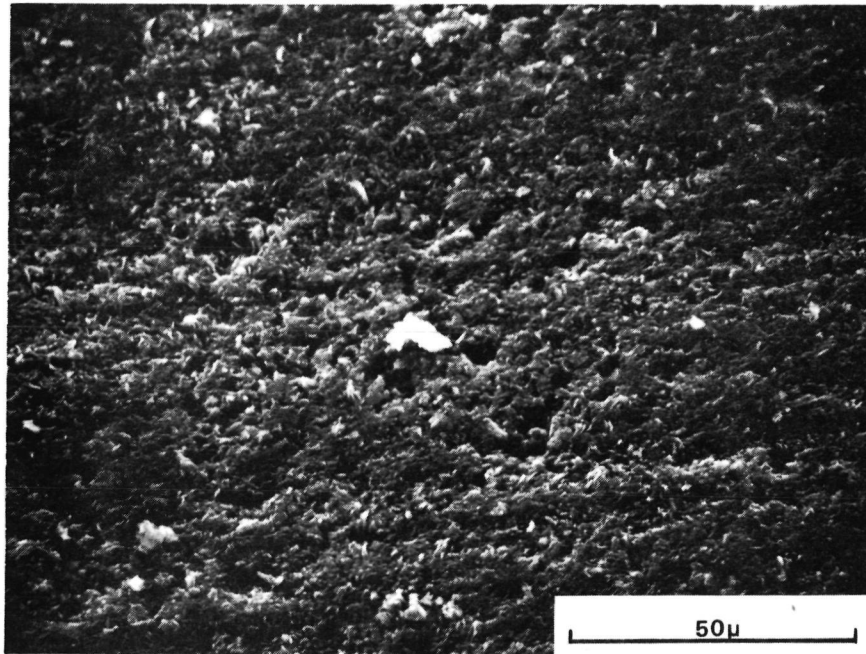
Graphite, Normal Incidence

Samples of C (high-density graphite) were sputtered with an etch dose of 240 mA-min/cm^2 using a S-S shield and an etch dose of 200 mA-min/cm^2 was also used with a S-S shield and CF_4 as the working gas. The nonsputtered control surface is shown in Fig. 4-7(a).

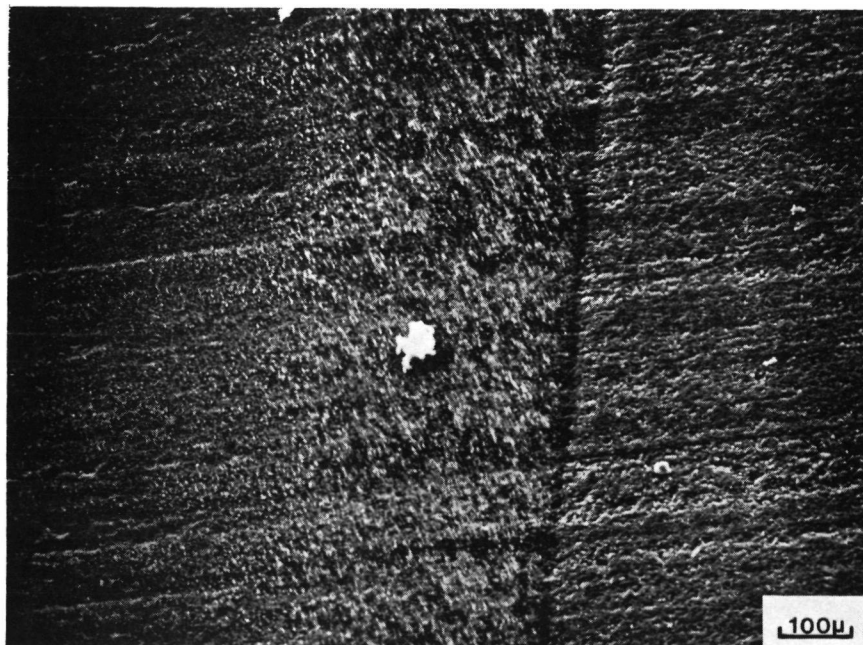
The transition between nonsputtered (right) and sputtered regions (left) is shown in Fig. 4-7(b). Even at the low magnification used, many whiskers are evident on the sputtered side. The length of the whiskers is greatest close to the shield edge, indicated by the straight line to the right of center. The whisker length decreases rapidly moving away from the shield edge, so that seeding from the shield is probably involved in the whisker growth. Figures 4-7(c) and (d) are higher magnifications of the long-whisker region near the center of Fig. 4-7(b). Figures 4-7(e) and (f) are higher magnifications of a region farther from the shield edge, and clearly show the shorter whiskers in that location.

The estimated etch depth of the C was about 1 micron, yet whiskers with lengths many times this depth were observed. It is therefore clear that the whiskers were the result of a "growth" process rather than simple material removal. A side view of the whiskers is shown in Fig. 4-7(g). The longest whisker (slightly to the left of center) is about 50 microns. Almost all the whiskers were normal to the surface. The few exceptions were believed broken in handling.

Figure 4-7(h) shows another sample with a Mo shield. The whiskers were also present with this sample, but much shorter. Also, there was a much smaller variation in length near the shield edge.



(a) Nonsputtered control surface.

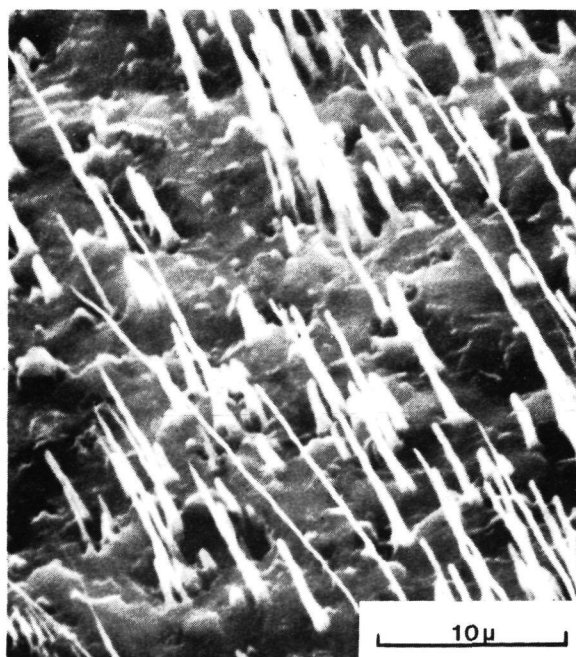


(b) Sputtered, Ar used as the working gas. Stainless steel shield, with etch dose of 240 mA-min/cm^2 .

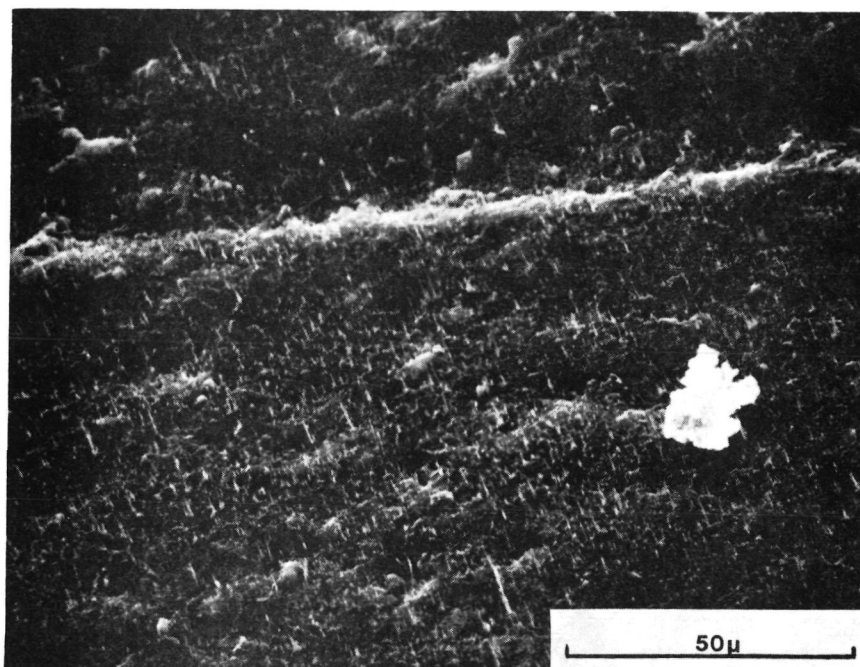
Figure 4-7 Graphite samples. Sputtering was at normal incidence.



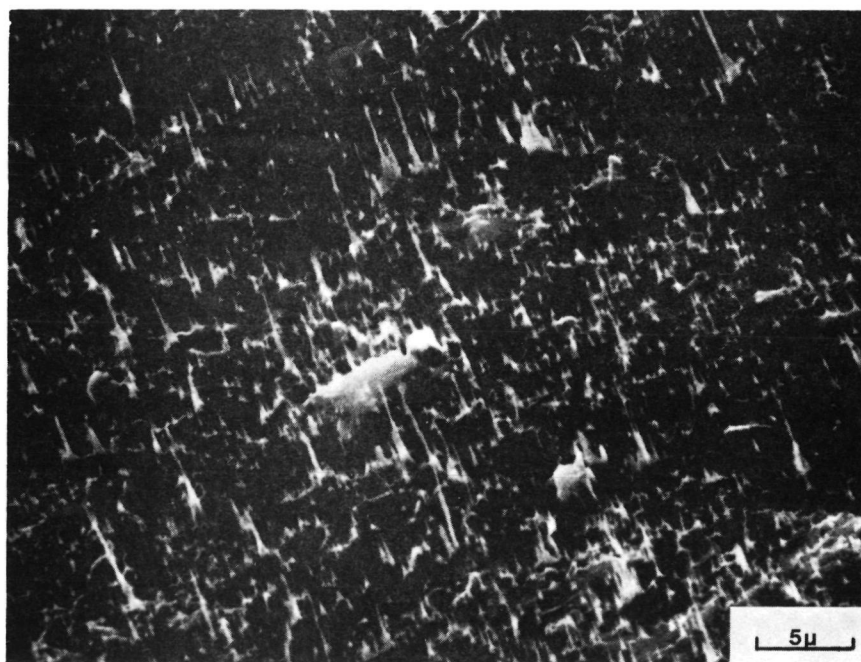
(c) Sputtered, Ar used as the working gas. Stainless steel shield, with etch dose of 240 mA-min/cm^2 .



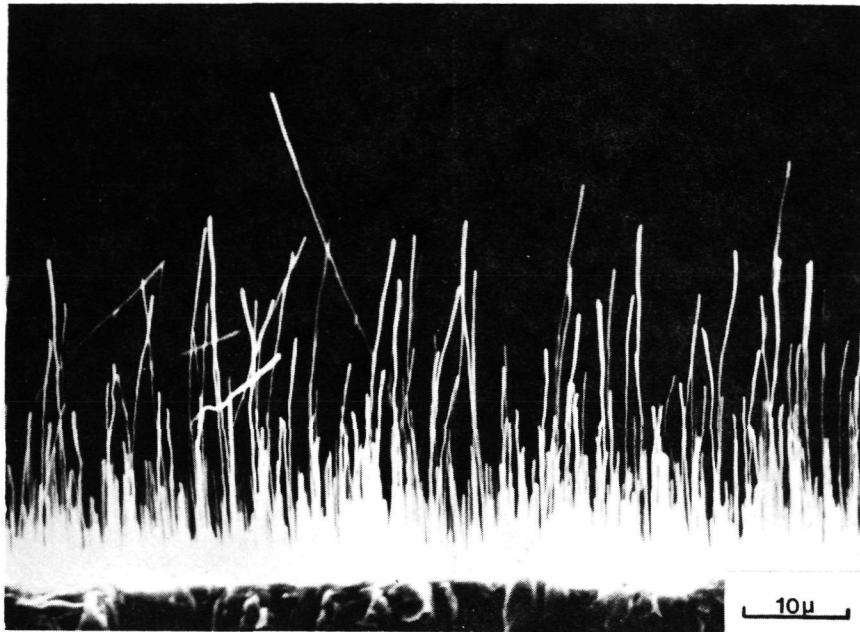
(d) Sputtered, Ar used as the working gas. Stainless steel shield, with etch dose of 240 mA-min/cm^2 .



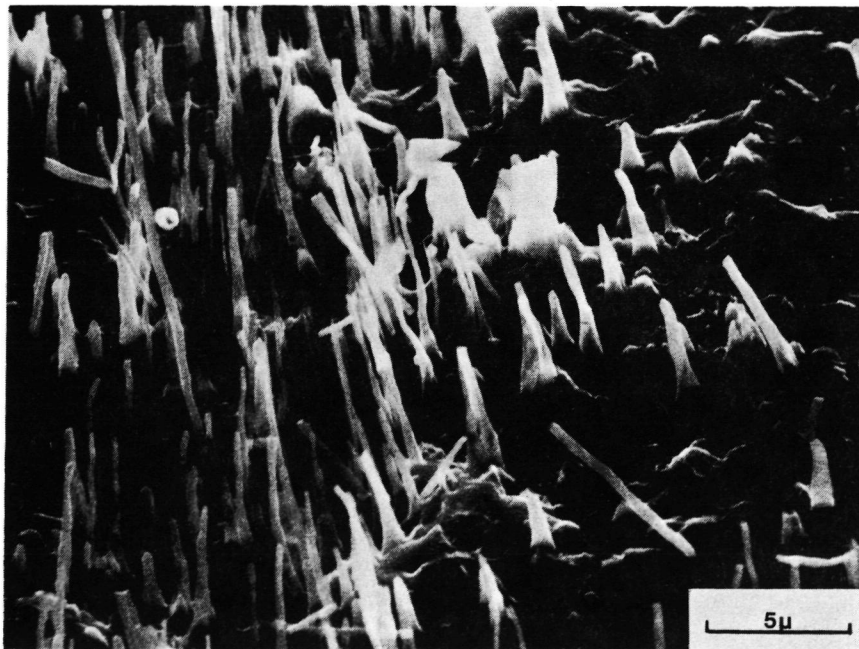
(e) Sputtered, Ar used as the working gas. Stainless steel shield, with etch dose of 240 mA-min/cm^2 .



(f) Sputtered, Ar used as the working gas. Stainless steel shield, with etch dose of 240 mA-min/cm^2 .

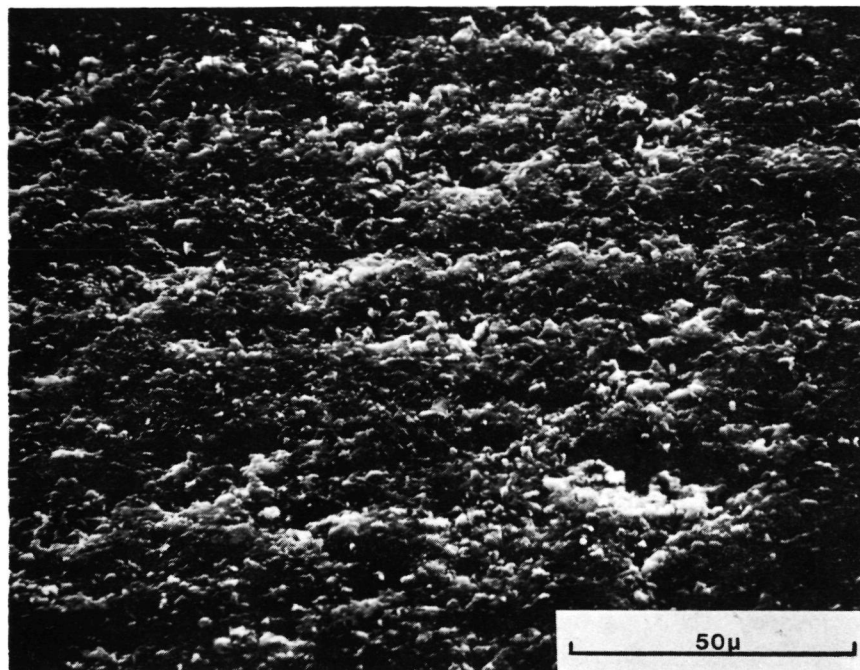


(g) Sputtered, Ar used as the working gas. Stainless steel shield, with etch dose of 240 mA-min/cm^2 .



(h) Sputtered, Ar used as the working gas. Molybdenum shield, with etch dose of 200 mA-min/cm^2 .

Figure 4-7(i) shows the sample etched using CF_4 . No whiskers were evident and the sample closely resembled the control sample shown in Fig. 4-7(a).



- (i) Sputtered, CF_4 used as the working gas. Stainless steel shield, with etch dose of 20 mA-min/cm^2 .

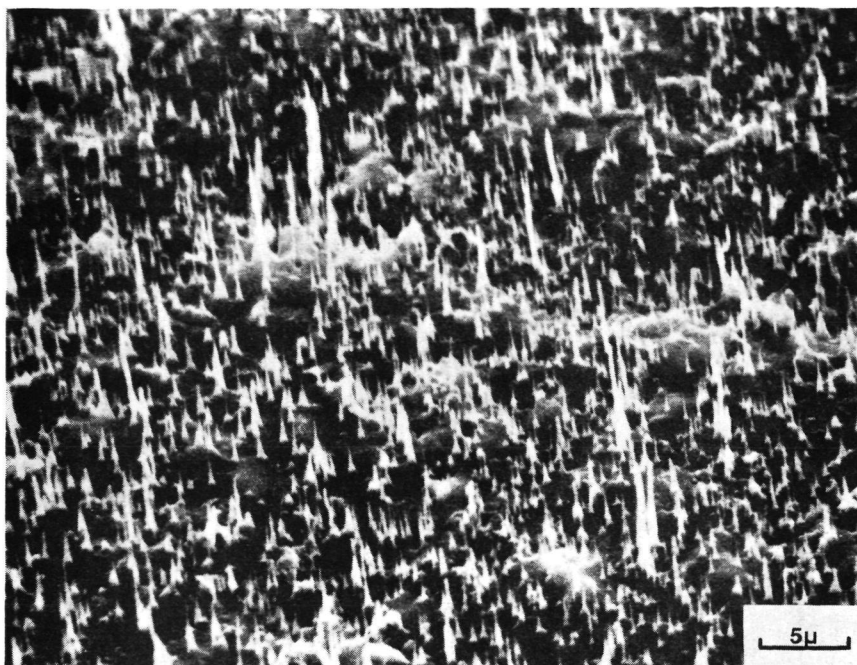
Graphite Variable Incidence

Samples of C were also sputtered at variable incidence angles. The etch dose was 200 mA-min/cm^2 using S-S shields and Ar as the working gas. The incidence was varied in 5° increments using sample holders similar to that shown in Fig. 4-1. Figure 4-8(a) shows a sample sputtered at normal incidence. The region shown is far from the shield. As might be expected, Fig. 4-8(a) closely resembles Fig. 4-7(f), which was of another sample etched under similar conditions.

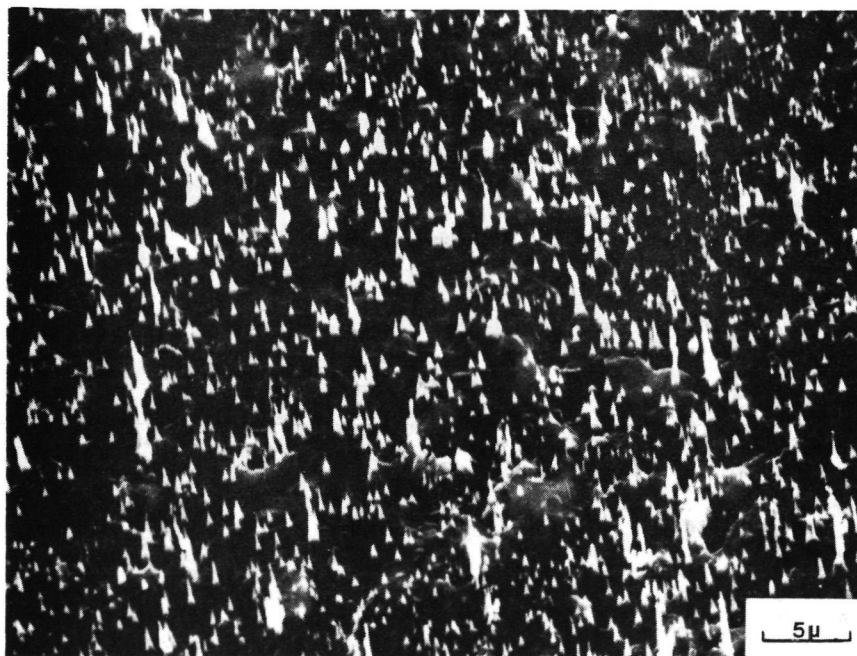
Fig. 4-8(b) is a sample etched at 10° from normal incidence, but appears similar to the sample etched at 5° . The whisker height is clearly reduced from that of the nontilted sample shown in Fig. 4-8(a). In fact, many of the whiskers are so short that they are cone-like.

Figure 4-8(c) is of a sample etched at 20° from normal, but is quite similar to micrographs of samples etched at 15° and 25° . The whiskers here appear thinner and longer than those obtained at 5° and 10° (Fig. 4-8(b)), but still shorter than those obtained at normal incidence (Fig. 4-8(a)).

A sample was also sputtered at 30° , Fig. 4-8(d), but no whiskers were obtained. At 35° , Fig. 4-8(e), the whiskers reappeared. The density of whiskers was much smaller at 35° than at normal incidence, but some were very long. At first glance, the long whiskers in Fig. 4-8(e) may appear due to contamination in handling (after the sputtering). But a closer examination of the micrograph will show that most, or all, of the long whiskers are attached to the sample. Further increases of angle to 40° , 45° , and 50° resulted in no whiskers, as indicated by Fig. 4-8(f), which was for 45° .

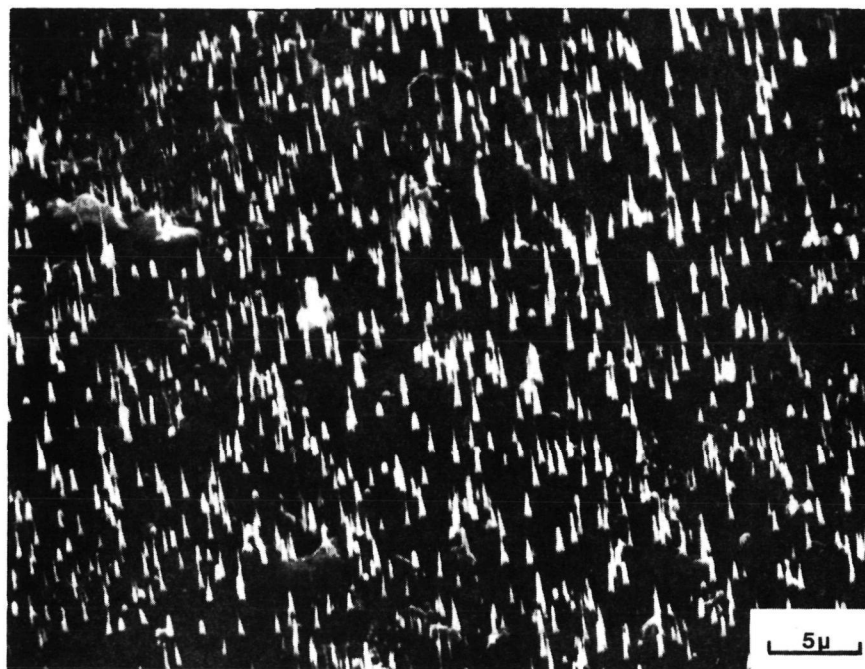


(a) 0° tilt (normal incidence).



(b) 10° tilt.

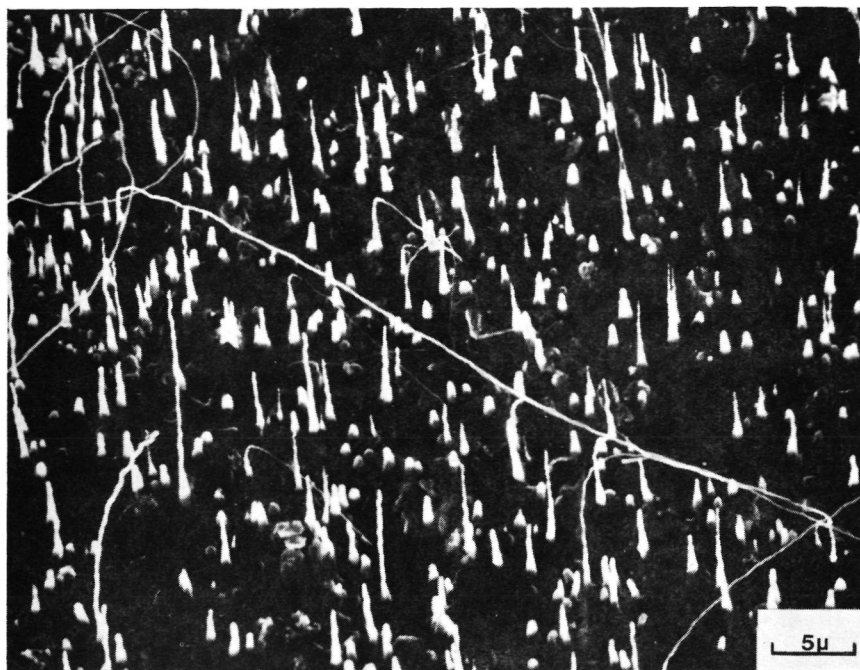
Figure 4-8 Graphite samples. Sputtering was at variable incidence, Ar used as the working gas. All with stainless steel shields and etch doses of 200 mA-min/cm^2 .



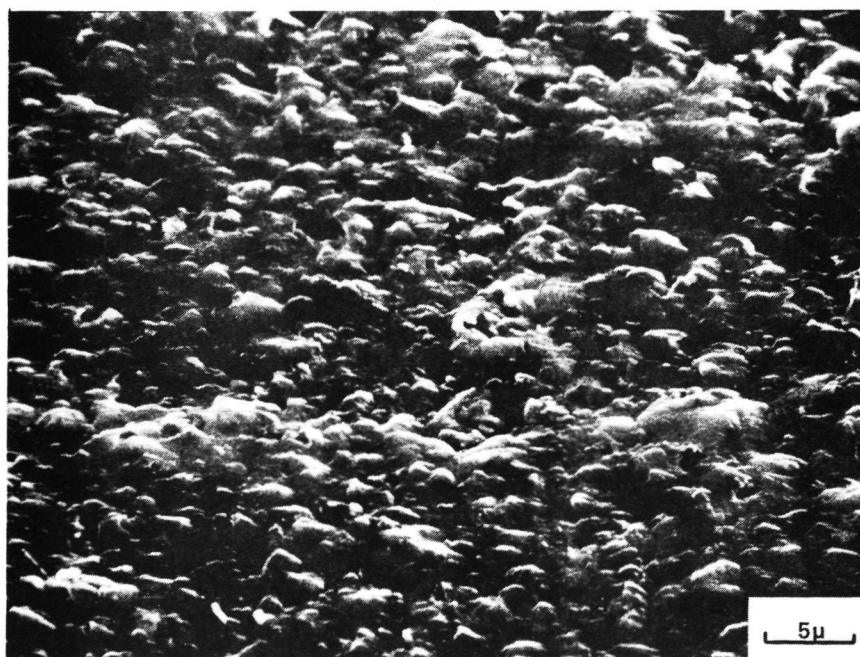
(c) 20° tilt.



(d) 30° tilt.



(e) 35° tilt.



(f) 45° tilt.

The angle of incidence for beam ions is clearly varied by changing the sample angle. There is also an electric field at the surface of the sample due to the plasma sheath that forms at that location. The electric field will always be normal to the sample surface because the sheath thickness is much smaller than the sample length and width. Because tilting the sample changes the relative incidence of the beam ions, but not the electric field, one might expect the growth of whiskers to be an angle-sensitive process. This angle sensitive nature of the growth process is, indeed, shown by Figs. 4-8(a) through (f). The nature of the growth process, however, appears quite complex. Hence, no attempt will be made to explain the sequence of events that were observed as the angle of incidence was varied.

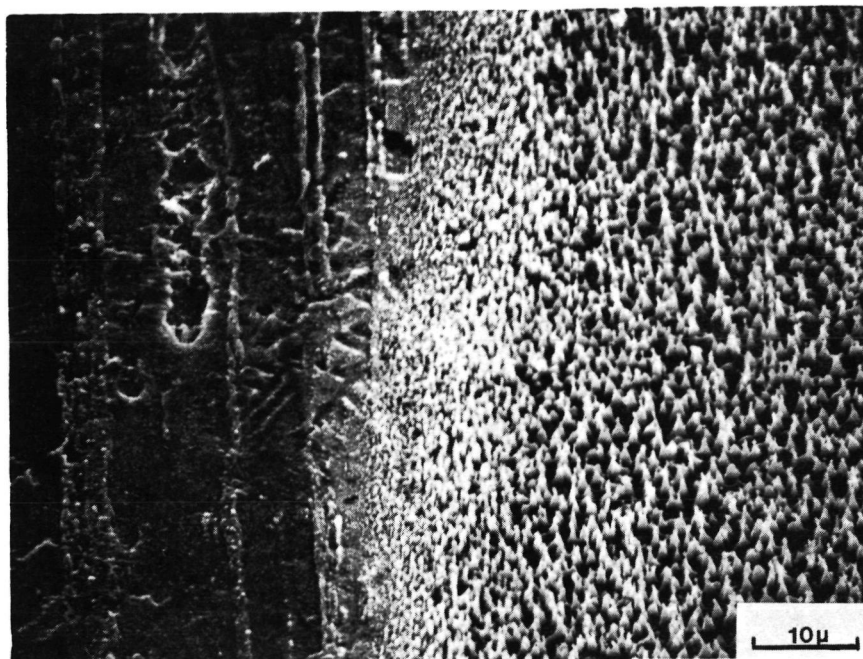
Copper

Samples of Cu were sputtered using Ar as the working gas with an etch dose of 240 mA-min/cm^2 (S-S shield) and with an etch dose of 200 mA-min/cm^2 (Mo shield, Ta shield, and no shield). The sample with no shield was sputtered at a 45° tilt, the rest were sputtered at normal incidence. Samples of Cu were also sputtered at normal incidence using CF_4 as the working gas with etch doses of 20 and 24 mA-min/cm^2 . The shields for these samples were S-S.

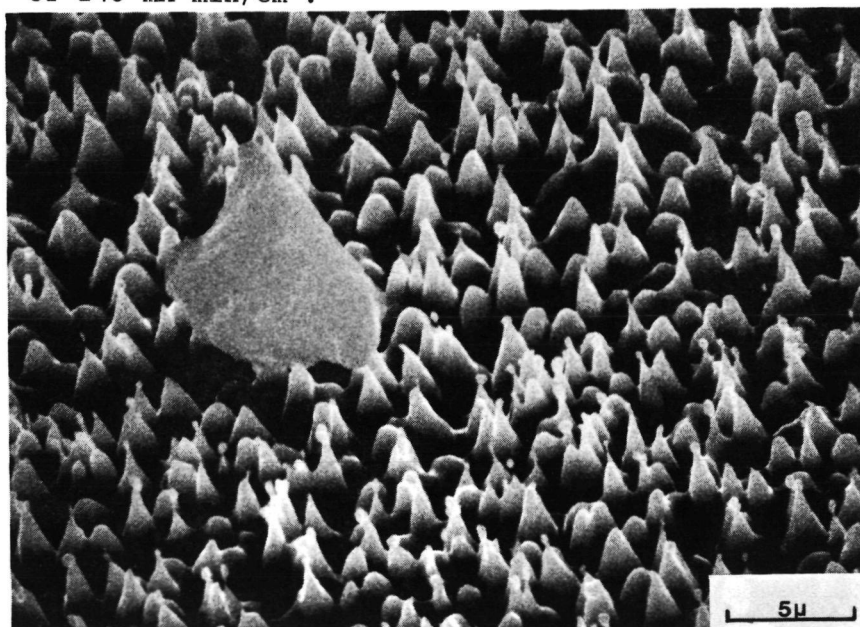
The transition from nonsputtered to sputtered regions is shown in Fig. 4-9(a) for a S-S shield and Ar. In contrast to the transition on a C sample, Fig. 4-7(b), this transition takes place in about 0.01 mm, beyond which there does not appear to be a significant change in cone density. A higher magnification of the cones well into the sputtered region is shown in Fig. 4-9(b). The cones appear to be several microns high. Because the cone height is small compared to the estimated etch depth of 20 microns for the etch dose used, this surface should be near a steady-state appearance and change only slightly with further etching.

Many of the cones formed using S-S (Figs. 4-9(a) and (b)) and Mo (Figs. 4-9(c) through (f)) shields have knobs attached to their apexes. It appears likely that these knobs are of low sputter yield impurities, hence, aid in the cone formation by protecting the apex. The sequence appears to be first the knob formation, then the eventual undercutting and removal of the knob, and finally the rounding of the apex. Cones in all phases of this sequence are visible in Figs. 4-9(b) and (d).

The transition from nonsputtered to sputtered regions is shown in Fig. 4-9(c) for a Mo shield and Ar. As with the S-S shield, this transition takes place in roughly 0.01 mm. Farther out, Fig. 4-9(d), the

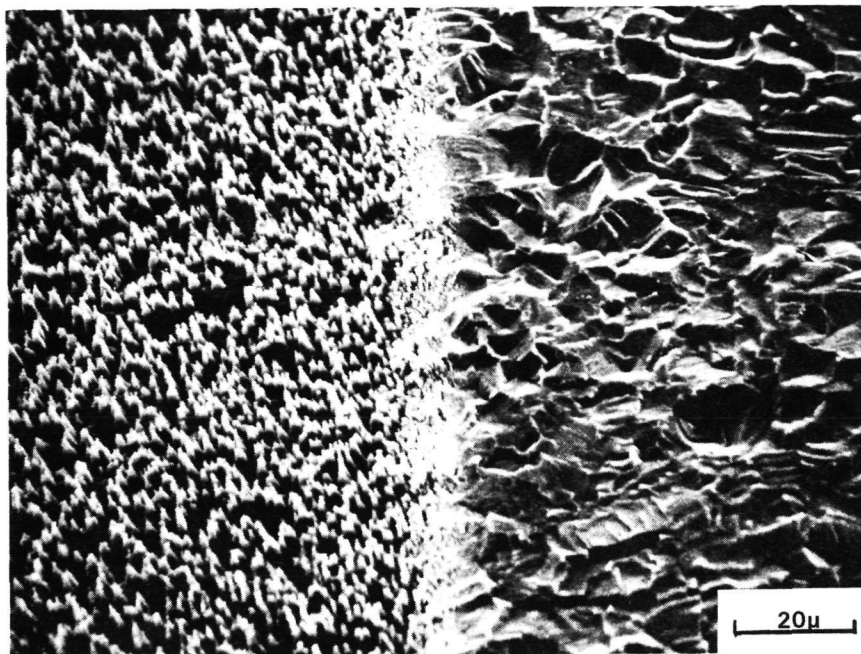


(a) Transition between nonsputtered (left) and sputtered regions. Sputtered, at normal incidence, Ar used as the working gas. Stainless steel shield, with etch dose of 240 mA-min/cm^2 .

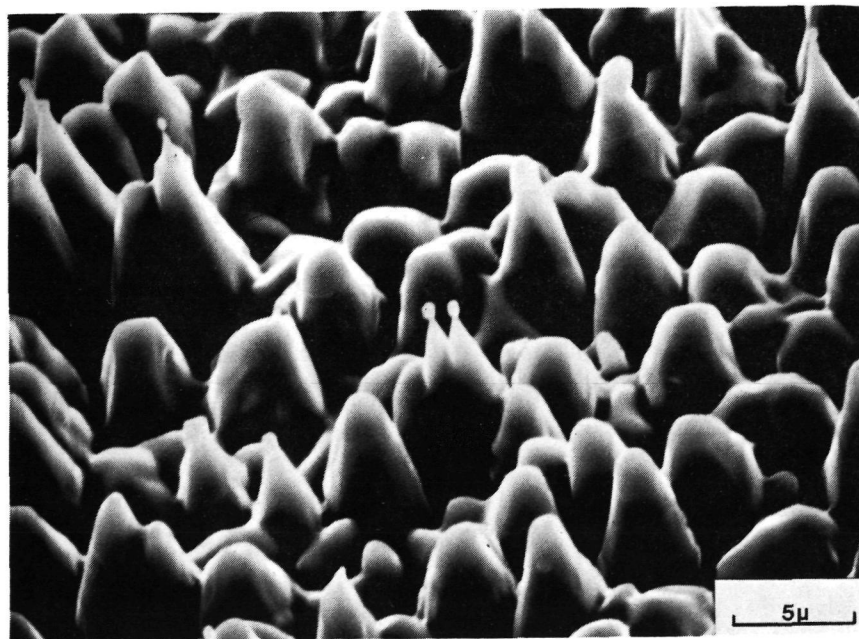


(b) Sputtered, at normal incidence, Ar used as the working gas. Stainless steel shield, with etch dose of 240 mA-min/cm^2 .

Figure 4-9 Copper samples.



(c) Transition between nonsputtered (right) and sputtered regions. Sputtered at normal incidence, Ar used as the working gas. Molybdenum shield, with etch dose of 200 mA-min/cm^2 .

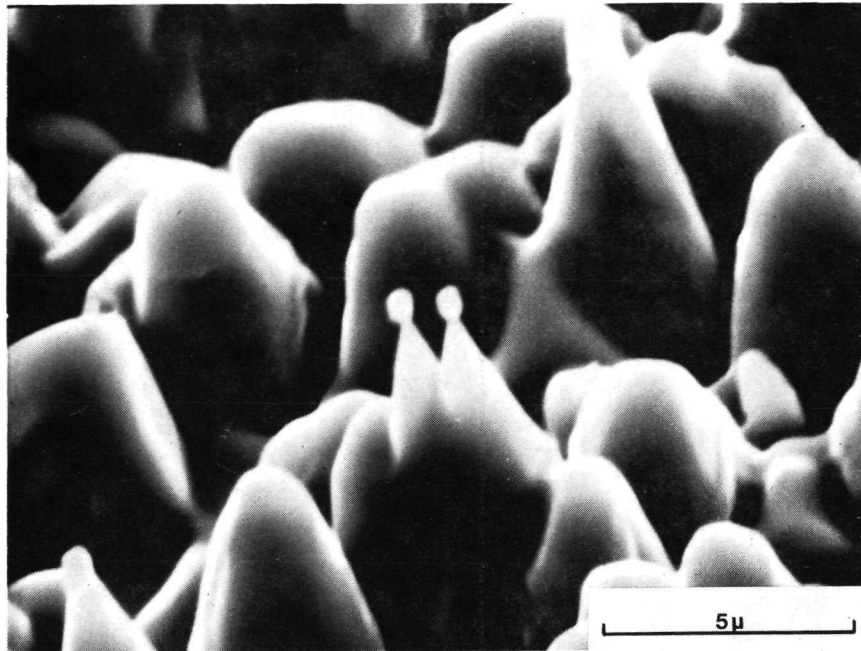


(d) Sputtered, at normal incidence, Ar used as the working gas. Molybdenum shield, with etch dose of 200 mA-min/cm^2 .

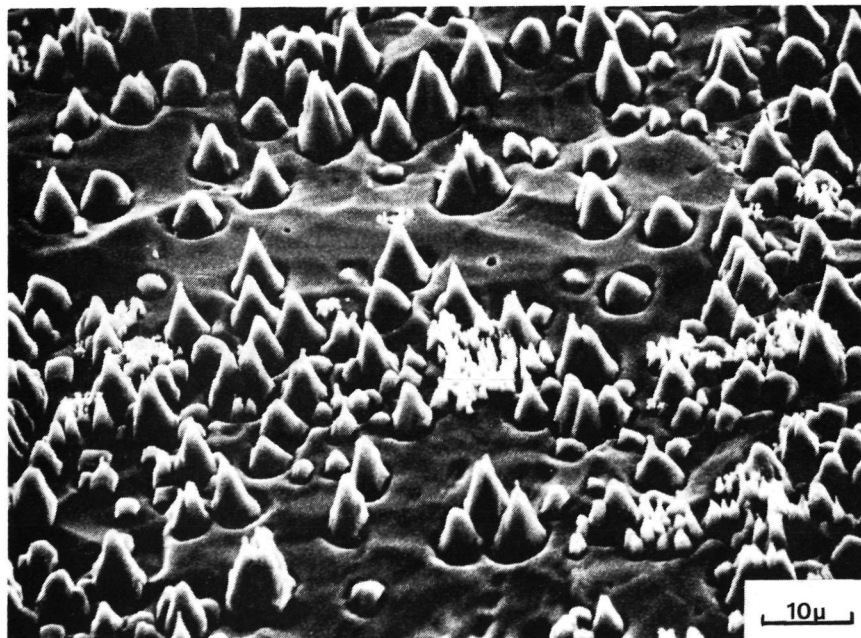
cones are similar to those with a S-S shield, but somewhat fewer in number. A further enlargement of a twin cone in Fig. 4-9(d) is shown in Fig. 4-9(e). Another sample with a Mo shield is shown in Fig. 4-9(f). There are even fewer cones in this micrograph, with some of the surface entirely free of cones. (The trenching around the base of the cones in Fig. 4-9(f) is a sputtering phenomenon normally found near the bases of inclined surfaces.) Both Fig. 4-9(d) and Fig. 4-9(f) were obtained several mm from the shield edge. The difference in these two micrographs is believed due to detailed differences in the shield edge, hence a difference in seeding.

The transition from nonsputtered to sputtered regions is shown in Fig. 4-9(g) for a Ta shield and Ar. This transition is more complex than that with either a S-S or Mo shield. Closest to the nonsputtered region is a narrow region similar to the ~ 0.01 mm transitions noted previously for Cu samples. Beyond this, however, is a strip 0.07-0.08 mm wide with a well-developed cone structure. This latter region is shown in a further enlargement in Fig. 4-9(h). Farther to the left in Fig. 4-9(g) is a finer etch pattern, shown also in the further enlargement of Fig. 4-9(i). This pattern is more complex than the cone structure of Fig. 4-9(h) and extends far from the shield edge. It appears from Figs. (a) through (i) that heavier seeding is required by Ta than by Mo and S-S for similar cone structures to be obtained.

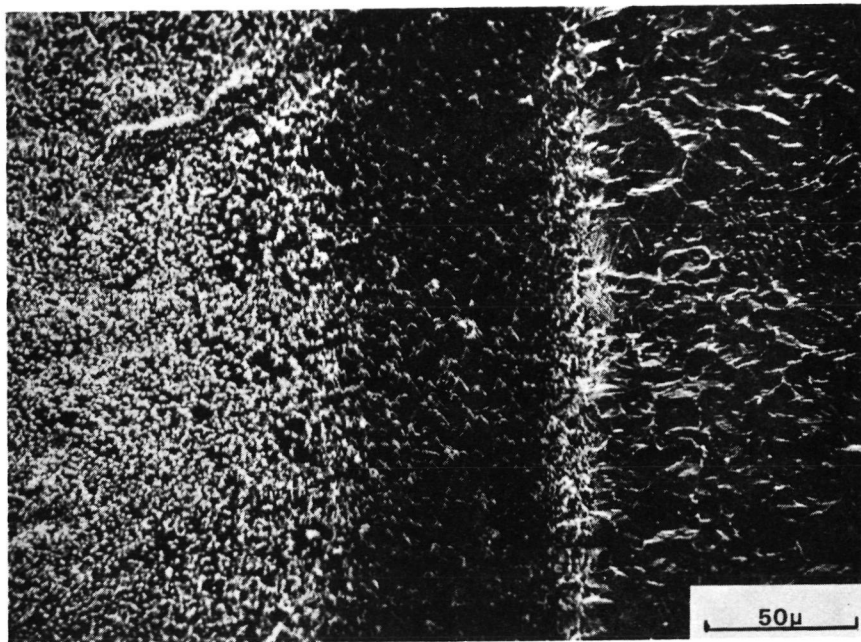
Visual inspection of all the Cu surfaces etched by a normal beam with Ar as the working gas usually revealed a dull reddish brown surface on the sputtered side, regardless of the shield material used. The only expectation to this appearance was found at certain inspection angles (probably dependent on cone angles) where a more shiny Cu tone was observed.



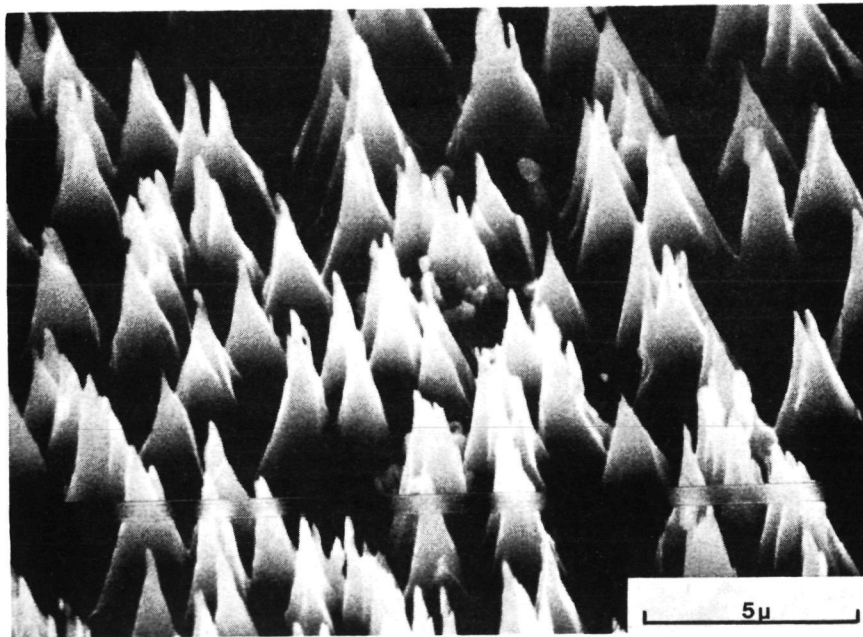
(e) Sputtered, at normal incidence, Ar used as the working gas. Molybdenum shield, with etch dose of 200 mA-min/cm².



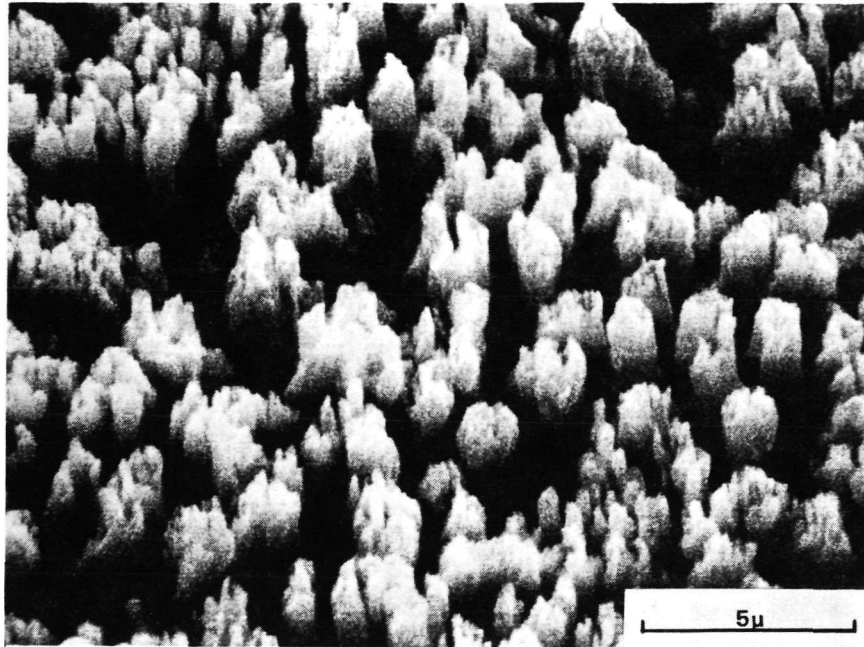
(f) Sputtered, at normal incidence, Ar used as the working gas. Molybdenum shield, with etch dose of 200 mA-min/cm².



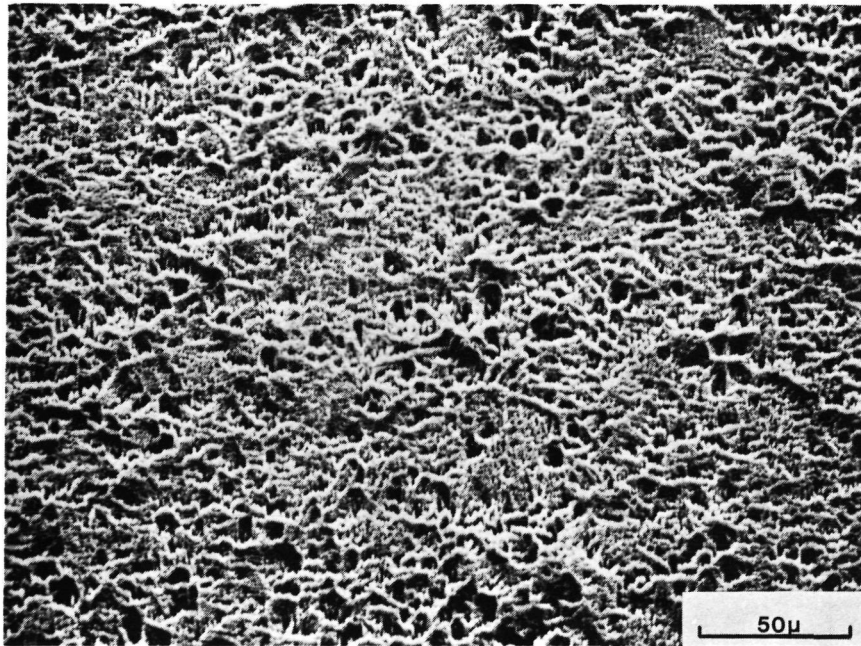
(g) Transition between nonsputtered (right) and sputtered regions. Sputtered, at normal incidence, Ar used as the working gas. Tantalum shield, with etch dose of 200 mA-min/cm^2 .



(h) Sputtered, at normal incidence, Ar used as the working gas. Tantalum shield, with etch dose of 200 mA-min/cm^2 .



- (i) Sputtered, at normal incidence, Ar used as the working gas. Tantalum shield, with etch dose of 200 mA-min/cm^2 .



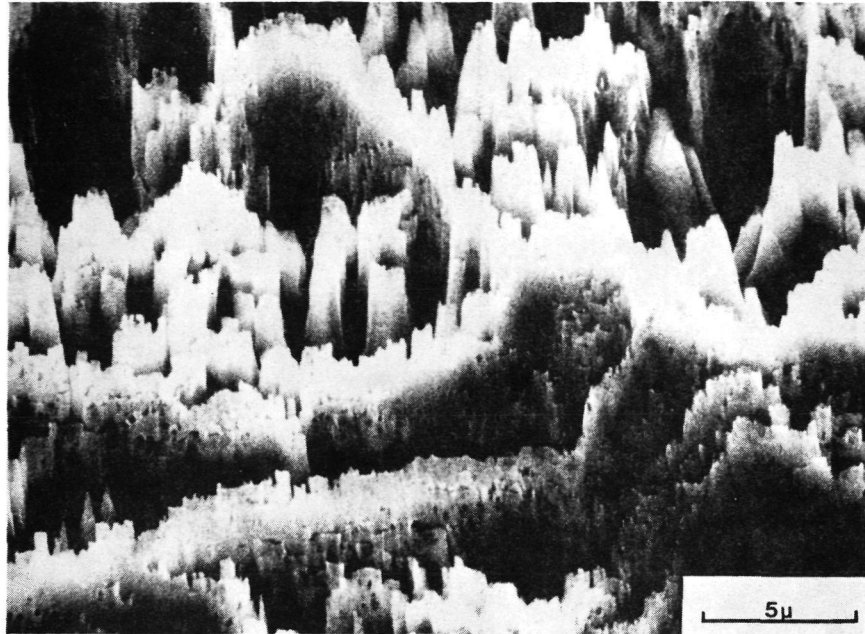
- (j) Sputtered, at 45° tilt, Ar used as the working gas. No shield, with etch dose of 200 mA-min/cm^2 .

Samples of Cu were also sputtered at a 45° tilt with Ar. No shields were used on these samples, but the support plate was S-S and could be "seen" by the sample. Seeding by S-S should therefore be assumed. The etching pattern obtained appears lacelike in Fig. 4-9(j). In the enlargement of Fig. 4-9(k), the pattern is seen to consist mostly of closely spaced rods of irregular cross section.

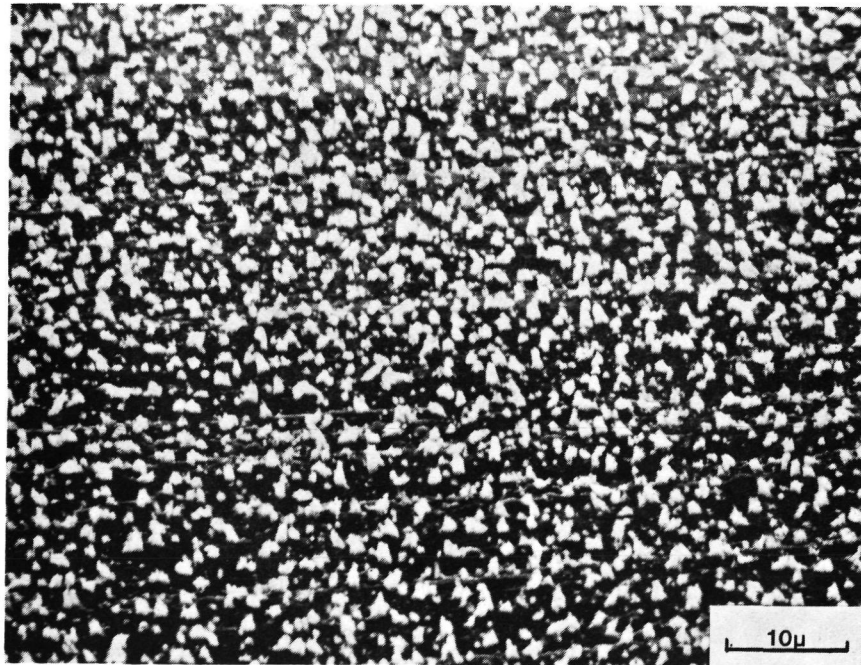
When viewed from the direction of the incident Ar ion beam, the 45° samples also exhibited a dull reddish brown appearance. When viewed from the angle of reflection (90° from the incident beam, but still 45° to the surface), the appearance was a bright S-S color. The latter observation strongly indicates heavy S-S seeding from the support plate.

A sample of Cu sputtered with CF_4 at normal incidence, using a S-S shield, is shown in Fig. 4-9(l). The pattern obtained consists of irregular shapes, up to 1 or 2 microns in size, that cover less than half the surface area. A further enlargement of this etch pattern is shown in Fig. 4-9(m). The pattern shown in these two micrographs extended over the entire sample surface with no significant change near the shield edge, which indicates seeding with S-S may not be important.

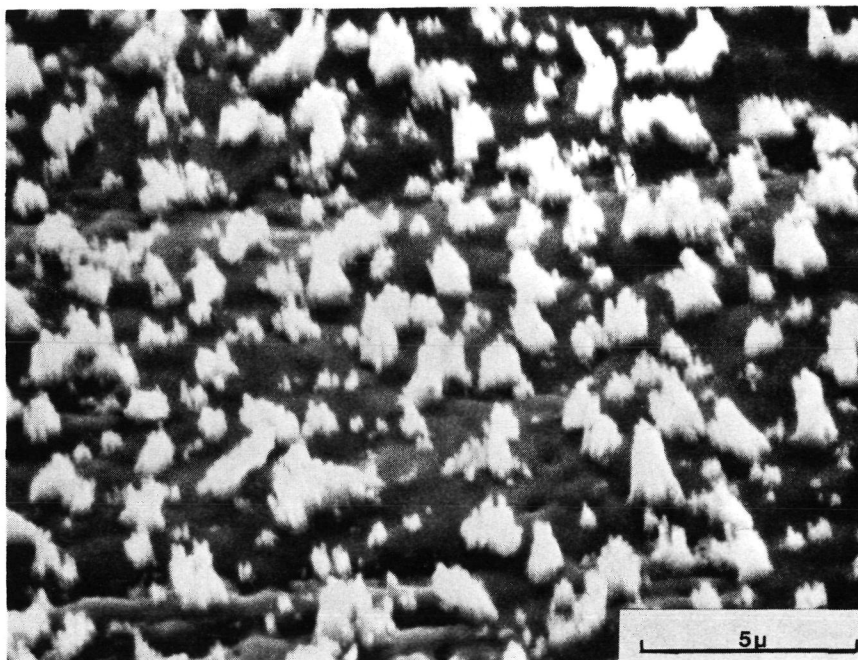
The sample of Cu shown in Figs. 4-9(l) and (m) were etched at an ion current density of 0.8 mA/cm^2 for 30 min., resulting in an etch dose of 24 mA-min/cm^2 . The sample shown in Fig. 4-9(m) was etched at an ion current density of 0.2 mA/cm^2 for 100 min., resulting in an etch dose of 20 mA-min/cm^2 . Inasmuch as the etch doses for Figs. 4-9(m) and (n) are nearly the same, one would expect the etch patterns to be nearly the same. Instead, Fig. 4-9(n) has a much finer pattern of irregular shapes covering a much higher fraction of the area. A possible reason for this difference in etch pattern is that etching with CF_4 as the



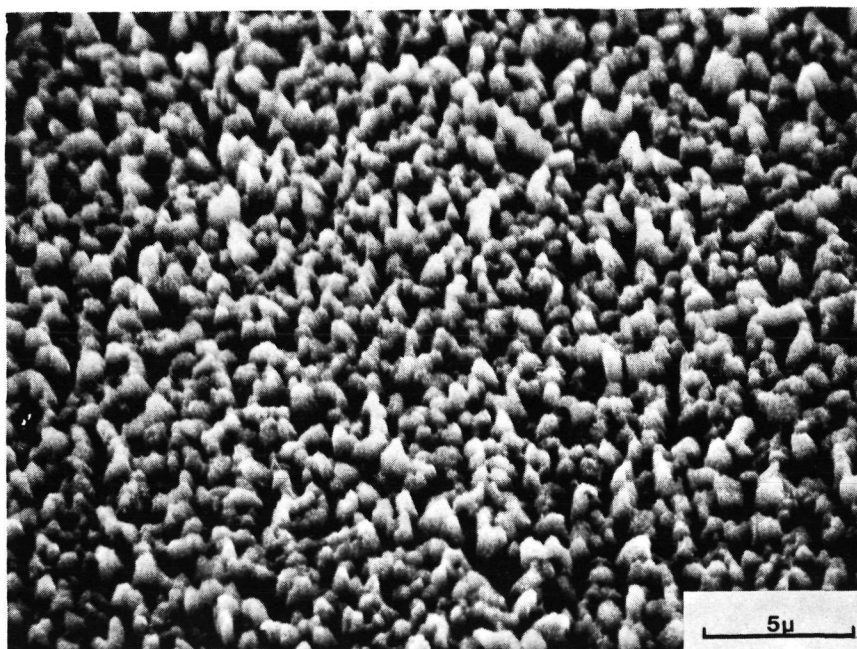
(k) Sputtered, at 45° tilt, Ar used as the working gas. No shield, with etch dose of 200 mA-min/cm^2 .



(l) Sputtered, at normal incidence, CF_4 used as the working gas. Stainless steel shield, with etch dose of 24 mA-min/cm^2 .



- (m) Sputtered, at normal incidence, CF_4 used as the working gas. Stainless steel shield, with etch dose of 24 mA-min/cm^2 .



- (n) Sputtered, at normal incidence, CF_4 used as the working gas. Stainless steel shield, with etch dose of 20 mA-min/cm^2 .

working gas involves chemical processes that may be rate dependent. An example of this rate dependence can be given in sputtering Si with CF_4 , which has been studied in plasma sputtering.¹ A small percentage of O_2 in the CF_4 will greatly increase the etch rate of Si, presumably because a protective layer of C that is otherwise deposited on the surface is thereby removed as CO or CO_2 . Because the pumping system was of the oil-diffusion type with only moderate cleanliness, a variety of background-impurity reactions might be possible.

The visual appearance of the Cu surfaces were changed very little by sputtering with CF_4 . A slight dullness was evident on the sputtered side when viewed from the proper angle.

Brass

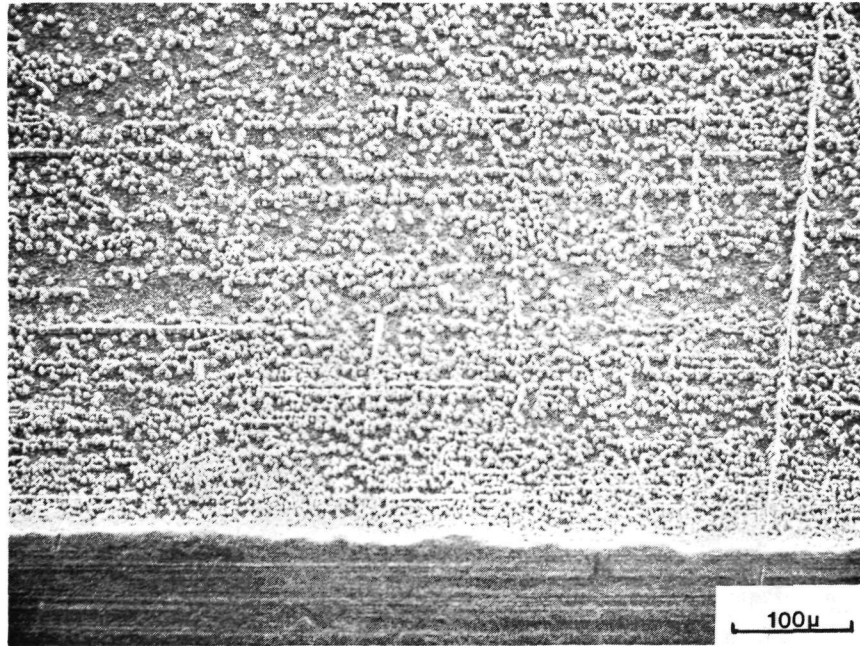
Samples of brass were sputtered with an etch dose of 200 mA-min/cm^2 using Ar as the working gas and an etch dose of 20 mA-min/cm^2 using CF_4 as the working gas. All samples used S-S shields. The micrograph of the nonsputtered control surface is shown in Fig. 4-10(a). The only major anomaly shown is in the upper right corner and is a suspected site of corrosion. This spot is small enough to miss during the cleaning procedure before sputtering.

The transition from nonsputtered (bottom) to sputtered (top) regions is shown in Fig. 4-10(b) for Ar at normal incidence. Some of the cone structures are seen to be concentrated along what appear to have been scratches before sputtering. The upper portion of Fig. 4-10(b) is representative of the entire sputtered surface of the sample. The cone structures are also shown in the further enlargement of Fig. 4-10(c). The pattern has a rough similarity to the Cu sample shown in Fig. 4-9(f). There are, however, no knobs visible in Fig. 4-10(c) and the surface not covered by the cones has a rougher appearance.

The sample of brass that was sputtered at a 35° tilt (to normal incidence) with Ar is shown in Figs. 4-10(d) through (f). A location close to the shield edge is shown in Fig. 4-10(d). A location well into the sputtered region is shown in Fig. 4-10(e), while Fig. 4-10(f) is near the edge opposite from the shield. These three micrographs are at about the same magnification, yet show three distinctly different etch patterns. These different patterns are presumably the result of the different seeding intensity of S-S. While there may have been some seeding from the S-S support plate, the amount of this seeding relative to that in Fig. 4-9(j) was probably reduced by both the smaller tilt

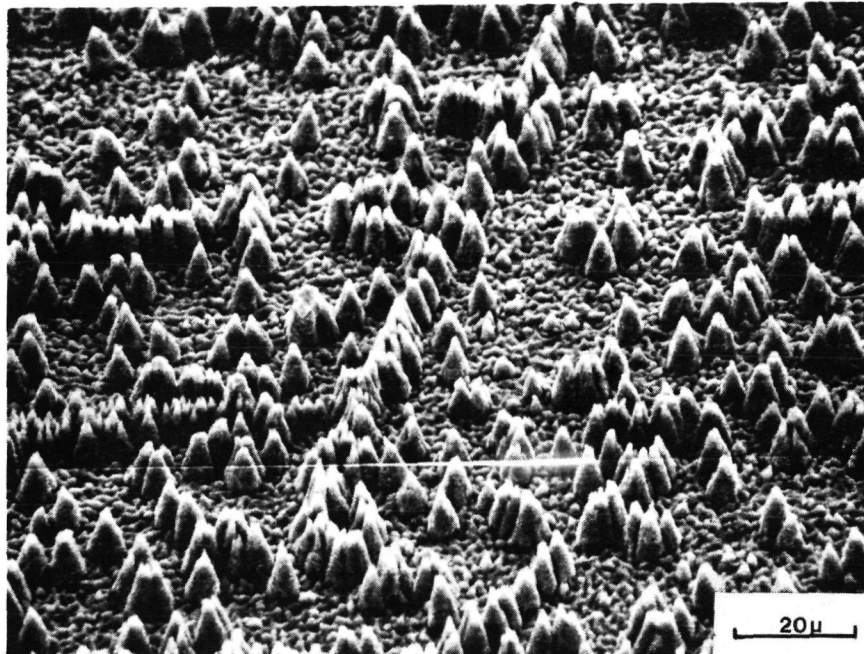


(a) Non-sputtered, control surface.

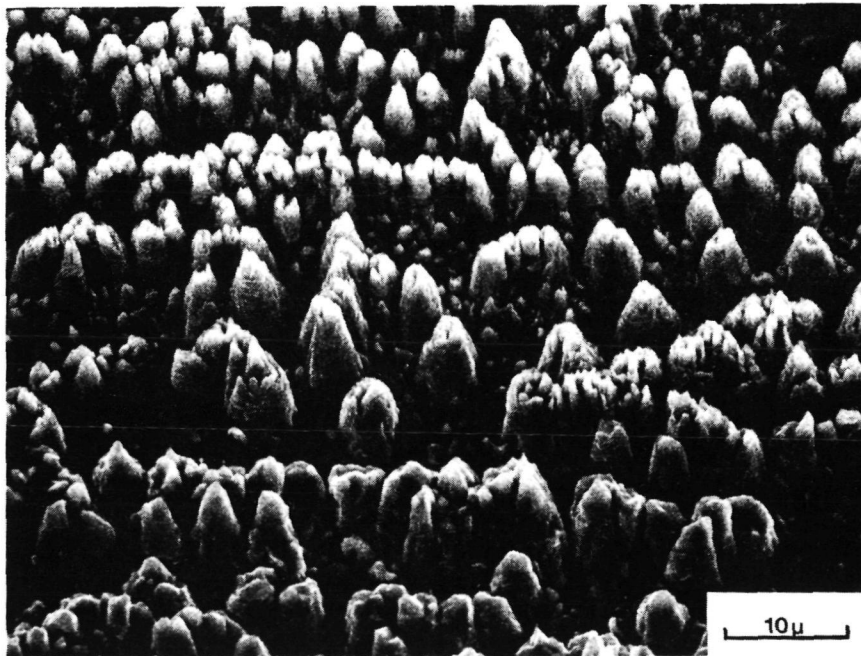


(b) Transition between non-sputtered (bottom) and sputtered regions. Sputtered at normal incidence, Ar used as the working gas, with etch dose of 200 mA-min/cm^2 .

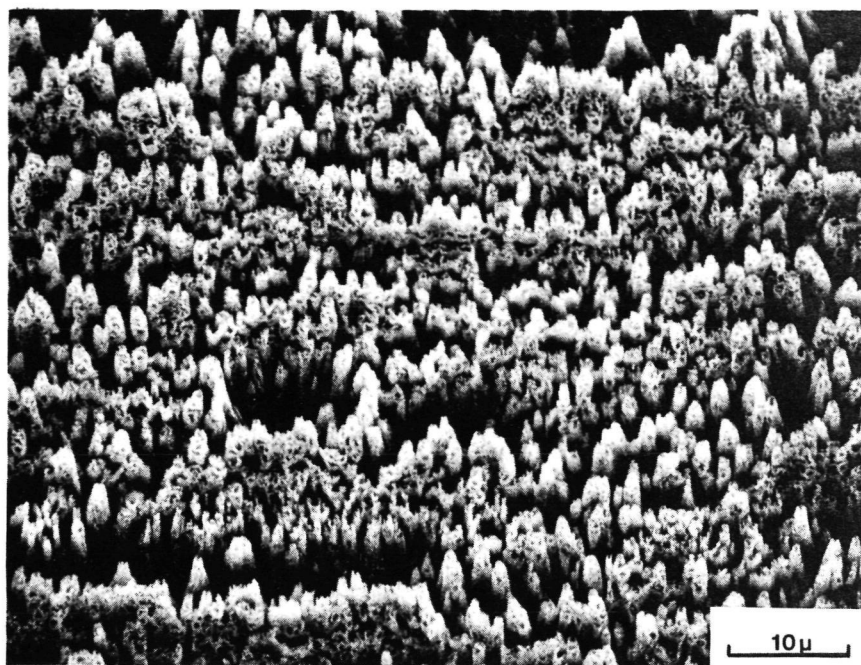
Figure 4-10 Brass samples. All with stainless steel shields.



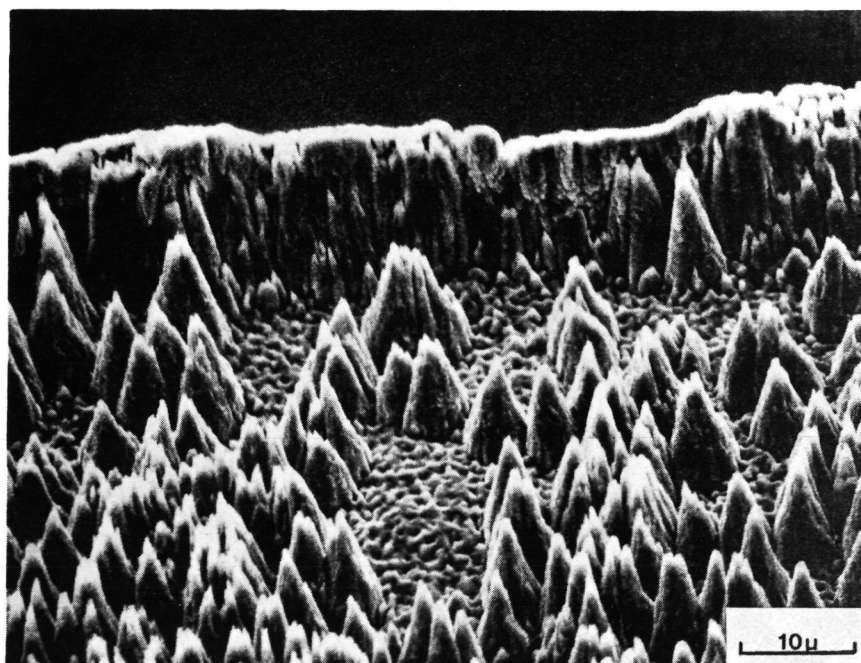
(c) Sputtered, at normal incidence, Ar used as the working gas. Etch dose of 200 mA-min/cm^2 .



(d) Sputtered, at 35° tilt (from normal), Ar used as the working gas. Etch dose of 200 mA-min/cm^2 .



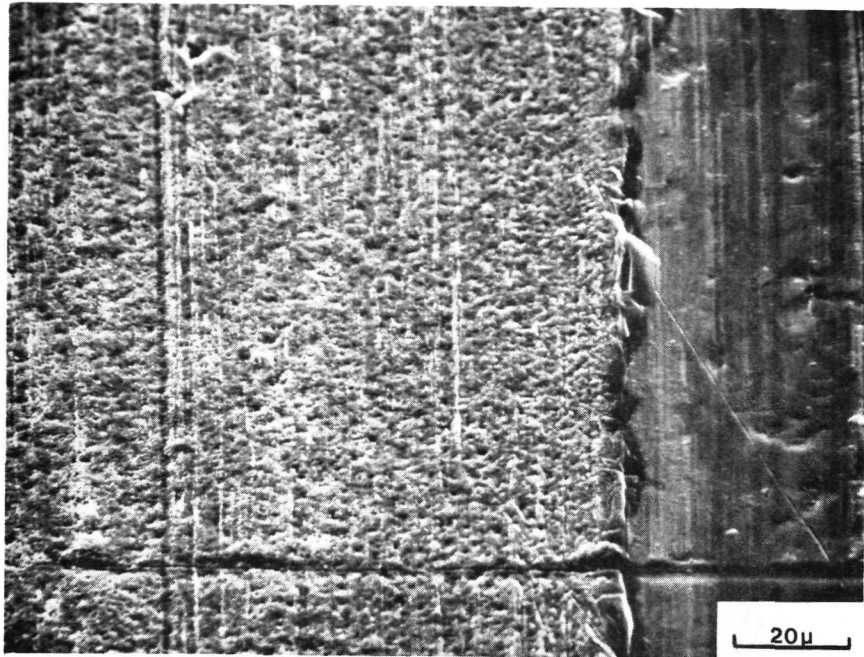
(e) Sputtered, at 35° tilt (from normal), Ar used as the working gas. Etch dose of 200 mA-min/cm^2 .



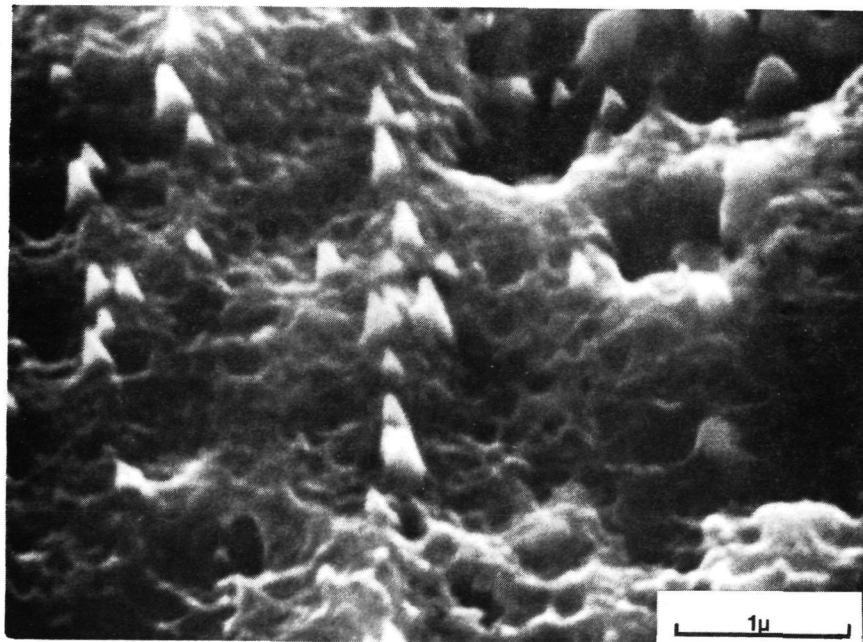
(f) Sputtered, at 35° tilt (from normal), Ar used as the working gas. Etch dose of 200 mA-min/cm^2 .

angle and the "shadowing" of the shield.

A sample of brass sputtered with CF_4 at normal incidence is shown in Figs. 4-10(g) and (h). The transition from nonsputtered to sputtered regions was very abrupt, as shown in Fig. 4-10(g). The etch pattern, shown in the future magnification of Fig. 4-10(h), was very uniform over the sputtered region. This pattern has a variety of interspersed features. There are scattered small cones on a rough surface. There are also small, nearly rectangular depressions. The small depressions might result from a high sputter yield constituent of the brass that had partially precipitated out in crystals. Visual inspection of the surface sputtered with CF_4 revealed a reddish brown color, probably due to enrichment of the Cu in the surface by preferential etching of another element or elements.



(g) Transition between non-sputtered (right) and sputtered regions. Sputtered, at normal incidence, CF_4 used as the working gas. Etch dose of 20 mA-min/cm².



(h) Sputtered, at normal incidence, CF_4 used as the working gas. Etch dose of 20 mA-min/cm².

V. ION BEAM INTERACTIONS

by Raymond S. Robinson

Effective application of ion sources to the various ion milling, reactive ion etching, surface texturing and sputter deposition processes requires characterization of the major interactions of the ion beam with the background environment through which it propagates. The major interaction processes are: (1) resonance charge exchange of fast ions with slow moving neutrals and (2) momentum exchange of fast moving neutrals with slow moving neutrals. A variety of other processes are involved but are quantitatively much less significant than these two.

For example, a small fraction of ions generated are doubly ionized. Depending on the ion source used and the operating conditions selected, the fraction of doubly ionized atoms extracted with the beam ranges from less than one percent to several percent.¹ In sputtering applications, double ions would strike the substrate with twice the energy of single ions. The effect of this doubly ionized fraction can be significant where depth of damage is critical, but is otherwise a negligible process.²

As another example, electrons are important for space-charge and current neutralization of the ion beam, but do not otherwise enter significantly into reactions. Electron masses are on the order of 10^{-5} of the ion or atom masses so that in the usual range of background densities the energy and momentum losses to electrons are negligible. In addition, the injection energy of electrons from a neutralizer is too low to cause significant ionization. The process of recombination of electrons with ions can likewise be neglected because the cross section for this

process ($\sim 10^{-9} \text{ \AA}^2$) is several orders of magnitude smaller than other cross sections of interest.³

As a final example, the momentum exchange of ions with background neutrals is relatively unimportant because: (1) the ion-atom collision cross section is somewhat less than for atom-atom collisions and (2) the charge exchange process has a much larger cross section than the momentum exchange process. The high velocity particles in the momentum loss process thus tend to be ions that have been charge exchanged to neutrals, rather than ions.

The background plasma through which the ion beam passes is largely the result of charge exchange between beam ions and the neutral background gas. In addition, neutral working gas flows out of the discharge chamber through the apertures in the accelerator system and produces a local density increase in the neutral background gas. There is another local density increase in the neutral background gas at the target due to beam ions that have given up their directed energy and have been neutralized by recombination with electrons at the target or substrate, thus contributing to the neutral gas background in the vacuum chamber. Aside from the local pressure maxima mentioned above, pressure in the vacuum chamber is governed by the speed of the pumping system relative to the flow rate of working gas into the discharge chamber.

Using known characteristics of practical ion sources, vacuum systems, and working gases some estimates can be made of the various deviations of actual beam properties from the ideal case of no beam-background interactions.

Charge Exchange

An ion and a neutral atom of the same element can undergo resonance charge exchange in which an electron is transferred from the atom to the ion e.g. $\text{Ar}_f^+ + \text{Ar} \rightarrow \text{Ar}_f + \text{Ar}^+$ (the subscript f indicates a fast moving particle relative to the second reactant). If a beam ion undergoes charge exchange with an atom of the background gas, the result is a slow ion and a fast neutral with essentially the original ion energy.

A functional form for the total cross section for resonance charge exchange is given by Iovitsu and Ionescu-Pallas⁴ and Rapp and Francis.⁵ Their expression for the total cross section for resonance charge exchange Q_r is

$$Q_r = (a - b \ln v)^2 \quad (5-1)$$

where a and b are constants to be determined using empirical data and v is the relative speed of the atom and ion. A curve of the form (5-1) was fit to experimental data from Dillon et. al.⁶ and Hasted⁷ using a least squares regression technique. The constants a and b were determined for argon resonance charge exchange:

$$a = 1.51 \times 10^{-9} \text{ m}$$

$$b = 9.53 \times 10^{-11} \text{ m}$$

Figure 5-1 is a plot of the total resonance charge exchange cross section for argon as a function of beam energy using this best fit for the function. At 500 eV, a typical ion beam energy, the total charge exchange cross section is about $23 \text{ \AA}^2$. The energy range plotted in Fig. 5-1 is extrapolated beyond the range of the experimental data used to fit the functional form, inasmuch as the actual data spanned the energy

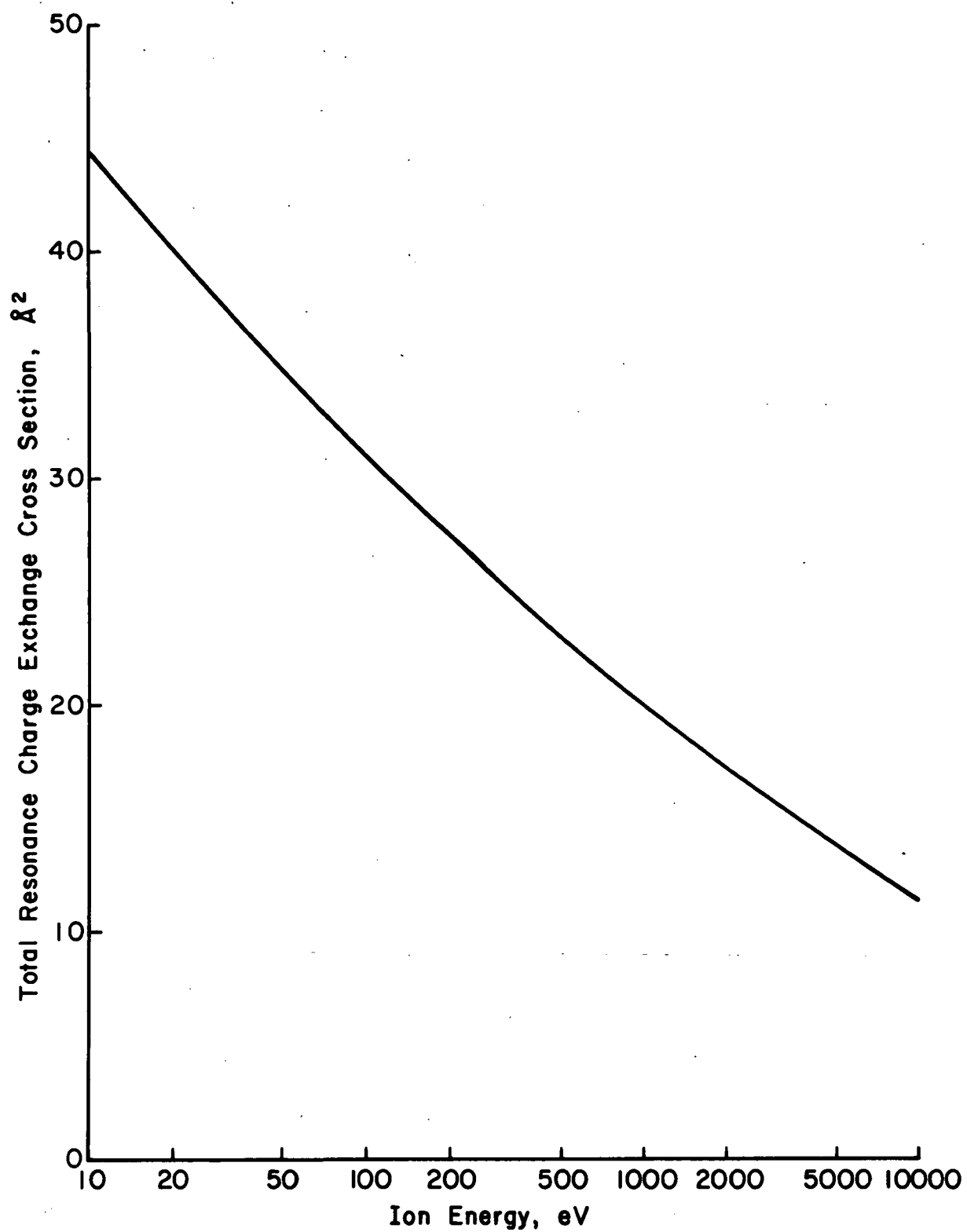


Figure 5-1. Total resonance charge exchange cross section for argon as a function of ion energy.

range from 50 eV to 850 eV. However, other experimental data compiled by Rapp and Francis⁵ are in good agreement with the extrapolated curve.

As the ion beam travels through the background gas in the vacuum chamber, the current in the beam as measured with a Faraday probe will show a decrease with distance from the ion source. (Because the current is the total integrated value, beam divergence is not a factor in this decrease.) This is because the ions are being neutralized by charge transfer and the probe will indicate only positive ions. The detection of slow ions is greatly reduced because they tend to migrate radially out of the beam shortly after charge transfer takes place. If resonance charge transfer were the only process to consider, the beam current, I , would decrease exponentially with distance x from the ion source.

$$I = I_0 e^{-nQ_r x} \quad (5-2)$$

where n is the number density of the neutral background gas and I_0 is the total beam current extracted from the ion source.

Because the density of fast neutrals in the beam can approach the density of the fast ions, the competing process of $Ar_f + Ar^+ \rightarrow Ar_f^+ + Ar$ should be considered. This inverse process has the same cross section Q_r , so that fast neutrals can be converted back to ions. However, the density of background ions is usually down by several orders of magnitude from the neutral density allowing the inverse process to be neglected.⁸

The mean free path λ of a beam ion with respect to charge transfer is given by

$$\lambda = \frac{1}{n Q_r} \quad (5-3)$$

Background pressures in a bell jar with an operating ion source typically range from 1×10^{-5} Torr to about 5×10^{-4} Torr, the corresponding range of mean free paths for a 500 eV ion beam would thus be roughly 13m down to 27cm. Operating in the upper end of this background pressure range would yield beams having a significant content of fast neutrals within a transport distance of a few tenths of a meter. Because beam current densities are customarily monitored using Faraday probes, accurate values for the total sputtering dose delivered to a substrate or target must take into account the neutral component of the beam.

Figure 5-2 is a plot of the product of the mean free path and the pressure as a function of ion energy. The curve was calculated from the data of Fig. 5-1 assuming ideal gas behavior at a temperature of approximately 300°K. At a given beam energy, dividing the ordinate by the background pressure yields a measure of the mean free path for resonance charge exchange.

A more refined estimate of the magnitude of the charge exchange effect could possibly be developed by considering the detailed effusion of neutral gas from the ion source rather than assuming a uniform density of background gas. Kaufman⁸ has considered the gradient of neutral density downstream of an ion truster and its effect on the generation of a charge exchange plasma. However, the accuracy of the experimental values used here for the charge exchange cross section probably does not warrant the inclusion of higher order effects in these estimates.

As an example of the loss in measured beam current with distance, the integrated beam current data taken using the 15cm multipole ion source (see Sec. III) can be compared with calculated values based on

the curve in Fig. 5-2. Table 5-1 compares total integrated beam currents at different distances from the ion source with values calculated for the two longer distances based on the value of the current at 5cm. Data to calculate the total integrated beam current at the ion source were unavailable; therefore, the current I_0 in Eq. (5-2) was calculated by inverting the equation and using the current measured 5cm from the source along with the appropriate cross section from Fig. 5-1. The source operating conditions were the same for all three current density measurements.

Table 5-1. Comparison of measured beam currents with values calculated from charge exchange considerations.

Distance from Source, cm	Pressure, Torr	Total Integrated Beam Current, ma	Calculated Beam Current, ma	Percent Difference
5	4×10^{-4}	98.7	—	—
10	4×10^{-4}	85.0	85.1	0.0%
15	2.8×10^{-4}	86.1	83.8	2.7%

The agreement between the calculated beam currents and the measured currents is seen to be excellent, supporting the charge exchange theory as summarized in Fig. 5-2.

Momentum Transfer

Only through scattering from particles of comparable mass can the fast particles of the beam lose significant energy and forward momentum. Scattering at sufficiently large angles will remove some ions or atoms entirely from the effective working volume of the beam. Because the neutral background gas has a density several orders of magnitude greater

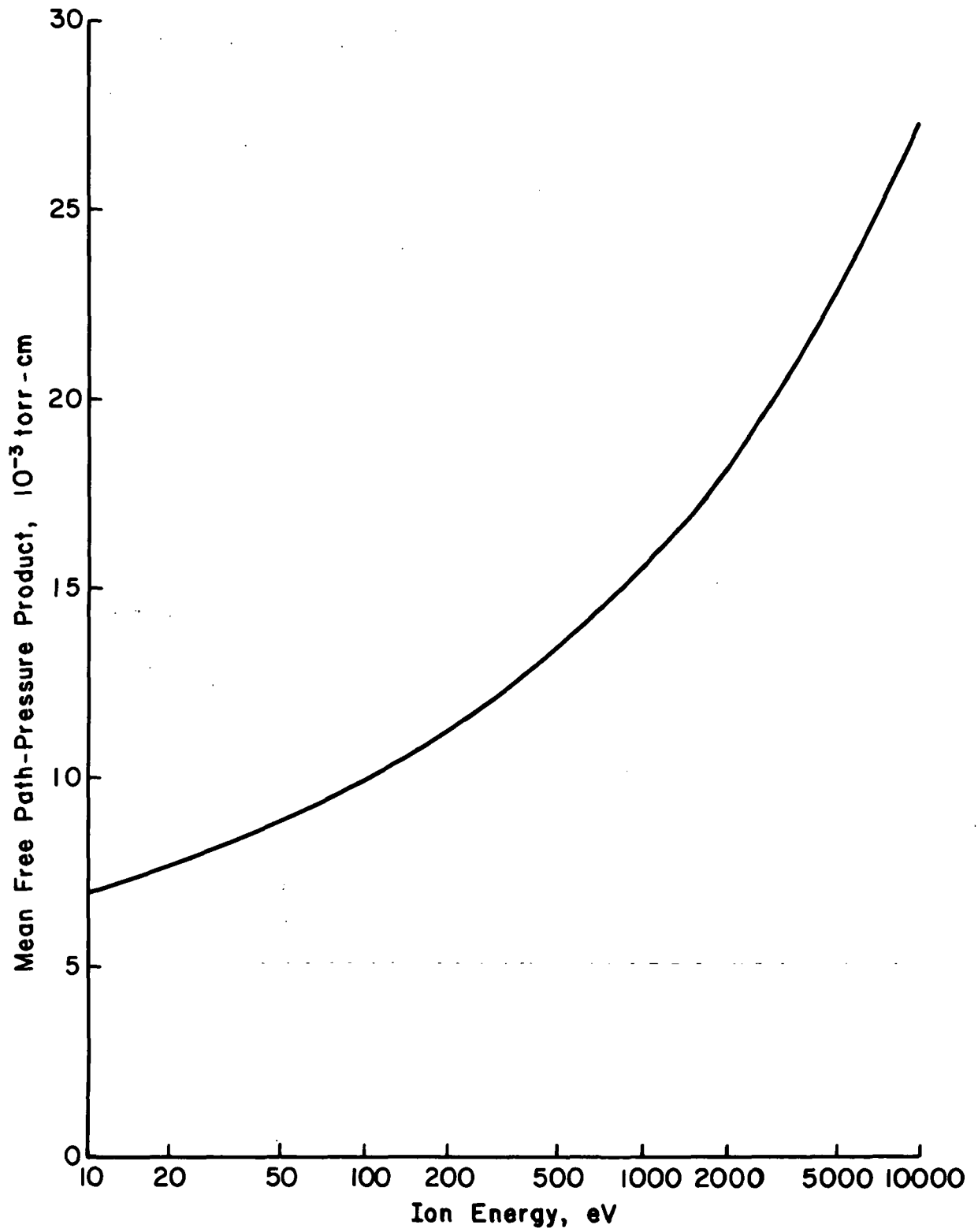


Figure 5-2. Argon resonance charge exchange: mean free path - pressure product as a function of ion energy.

than the background ion density, collisions with neutral atoms are dominant compared with collisions involving the background charge exchange ions. Further, the probability of charge exchange prior to elastic collisions is high for incident ions. The collisions of high velocity neutrals with low velocity neutrals is thus the dominant process for significant momentum and energy loss by sputtering particles.

Collision Dynamics

The deBroglie wavelength of an argon atom or ion with a typical beam energy of 500 eV is:

$$\frac{h}{p} = 2.03 \times 10^{-13} \text{ m}$$

where h is Planck's constant and p is the momentum of the atom. This wavelength is very small compared to atomic dimensions, allowing the use of classical particle trajectories as a first approximation to the scattering of argon from argon. Measuring the angular momentum of the two colliding atoms in units of $h/2\pi$ yields an angular momentum quantum number for the collision that is typically several thousand for impact parameters of the order of atomic dimensions. This is a further indication that classical dynamics is applicable since most collisions of interest fall in the realm of very large quantum numbers.

If the interaction potential between two atoms is known, the classical equations of motion can be solved to relate the scattering angle to the impact parameter for the incident atom. The solution is most directly obtained using center of mass coordinates where a particle of reduced mass μ interacts with a fixed center of force through a potential $V(r)$

where r is the separation distance of the particle from the center of force.⁹ Conservation of energy can be expressed as

$$\frac{1}{2} \mu v_0^2 = \frac{1}{2} \mu \dot{r}^2 + \frac{1}{2} \mu r^2 \dot{\phi}^2 + V(r) \quad (5-4)$$

where v_0 is the relative speed of the two atoms at large separations where $V(r)$ is negligible, and ϕ and r are the angular and radial coordinates respectively of the incoming particle.

Conservation of angular momentum can be expressed as

$$\mu v_0 s = \mu r^2 \dot{\phi} \quad (5-5)$$

where s is the impact parameter (see Fig. 5-3). Eliminating $\dot{\phi}$ between Eqs. (5-4) and (5-5) and using the chain rule for differentiation we can write:

$$\frac{dr}{d\phi} = \frac{r^2}{s} \left(1 - \frac{s^2}{r^2} - \frac{V(r)}{\frac{1}{2} \mu v_0^2} \right)^{\frac{1}{2}} \quad (5-6)$$

or,

$$d\phi = \frac{s}{r^2} \left(1 - \frac{s^2}{r^2} - \frac{V(r)}{\frac{1}{2} \mu v_0^2} \right)^{-\frac{1}{2}} dr \quad (5-7)$$

Integration of (5-7) gives the angle through which the radius vector turns during the collision. Let the total turning angle be ϕ_t , then

$$\phi_t = 2 \int_{r_0}^{\infty} \frac{s}{r^2} \left(1 - \frac{s^2}{r^2} - \frac{V(r)}{\frac{1}{2} \mu v_0^2} \right)^{-\frac{1}{2}} dr \quad (5-8)$$

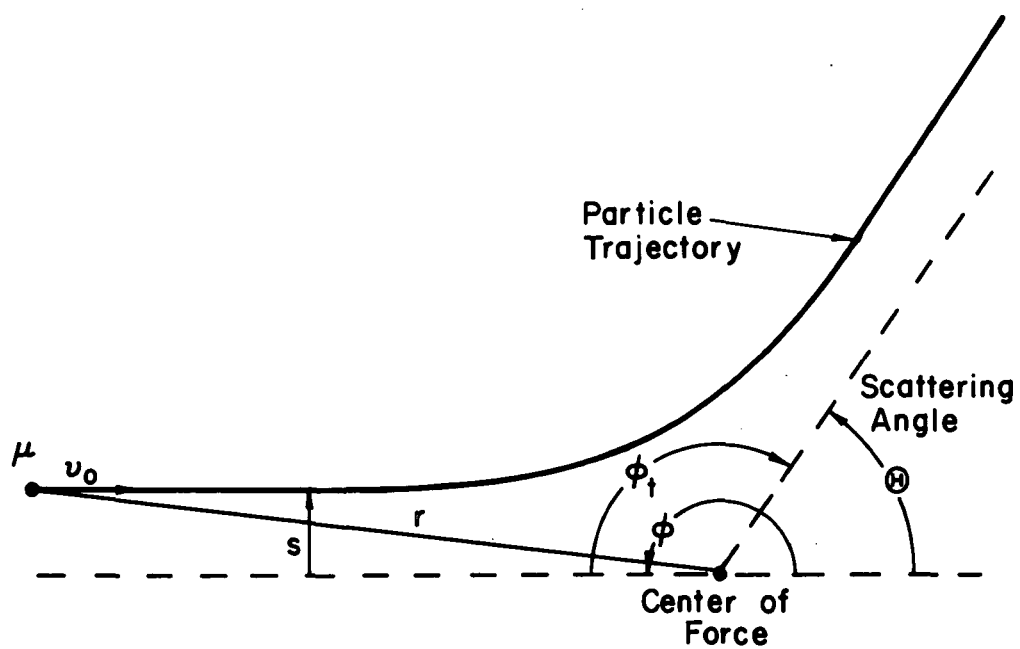


Figure 5-3. Scattering of reduced mass particle by a center of force.

where r_o is the distance of closest approach. The scattering angle in the center of mass system is given by

$$\Theta = \pi - \phi_t \quad (5-9)$$

Therefore,

$$\Theta = \pi - 2s \int_{r_o}^{\infty} \frac{dr}{r^2} \left(1 - \frac{s^2}{r^2} - \frac{V(r)}{\frac{1}{2}\mu v_o^2} \right)^{-\frac{1}{2}} \quad (5-10)$$

which relates the scattering angle in the center of mass system to the impact parameter and energy of the incident particle.

For incident and target particles having equal masses (which is the case of most interest), the scattering angle in the center of mass system is easily converted to the true scattering angle θ in the laboratory frame of reference:¹⁰

$$\theta = \frac{\Theta}{2} \quad (5-11)$$

The integration of Eq. (5-10) is not straightforward because the upper limit is infinite and the integrand itself has a singularity at the point of closest approach. Closed form solutions in terms of elliptic integrals are known for only a few power-law functions of r for the potential $V(r)$.⁹

The actual integration can be carried out numerically after a change of variables to $u = 1/r$ which gives finite limits but does not remove the singularity in the integral. An algorithm was developed for this integration utilizing a ten-point Gauss-Legendre quadrature scheme along with a logarithmically decreasing integration interval as the integration

proceeds from the well-behaved end of the interval toward the singularity. Before integrating, the upper limit of integration, $u_0 = 1/r_0$, was first determined by an iterative solution to the (usually) transcendental equation:

$$\frac{2V(r_0)}{E_t} + \frac{s^2}{r_0^2} = 1 \quad (5-12)$$

where E_t is the energy of the incident particle in the laboratory frame of reference.

To verify that the integration algorithm was functioning properly, scattering angles for an inverse square law force (Rutherford Scattering) were calculated. These calculated angles for 1eV argon ions incident on argon ions were compared with the closed-form solution, Table 5-2 and Fig. 5-4. The qualitative agreement between the numerical and closed form solutions is adequately demonstrated in Fig. 5-4, while the numerical values tend to exhibit a divergence of only a few units in the third significant figure.

Table 5-2. Comparison of numerical integration with closed form solution for Rutherford scattering.

Impact Parameter Å	θ , Closed Form deg.	θ , Numerical Integration deg.
10	55.22	55.05
20	35.75	35.59
30	25.64	25.39
40	19.80	19.57
50	16.07	15.61
60	13.50	13.49
70	11.62	11.63
80	10.20	10.13
90	9.09	9.06
100	8.19	8.20

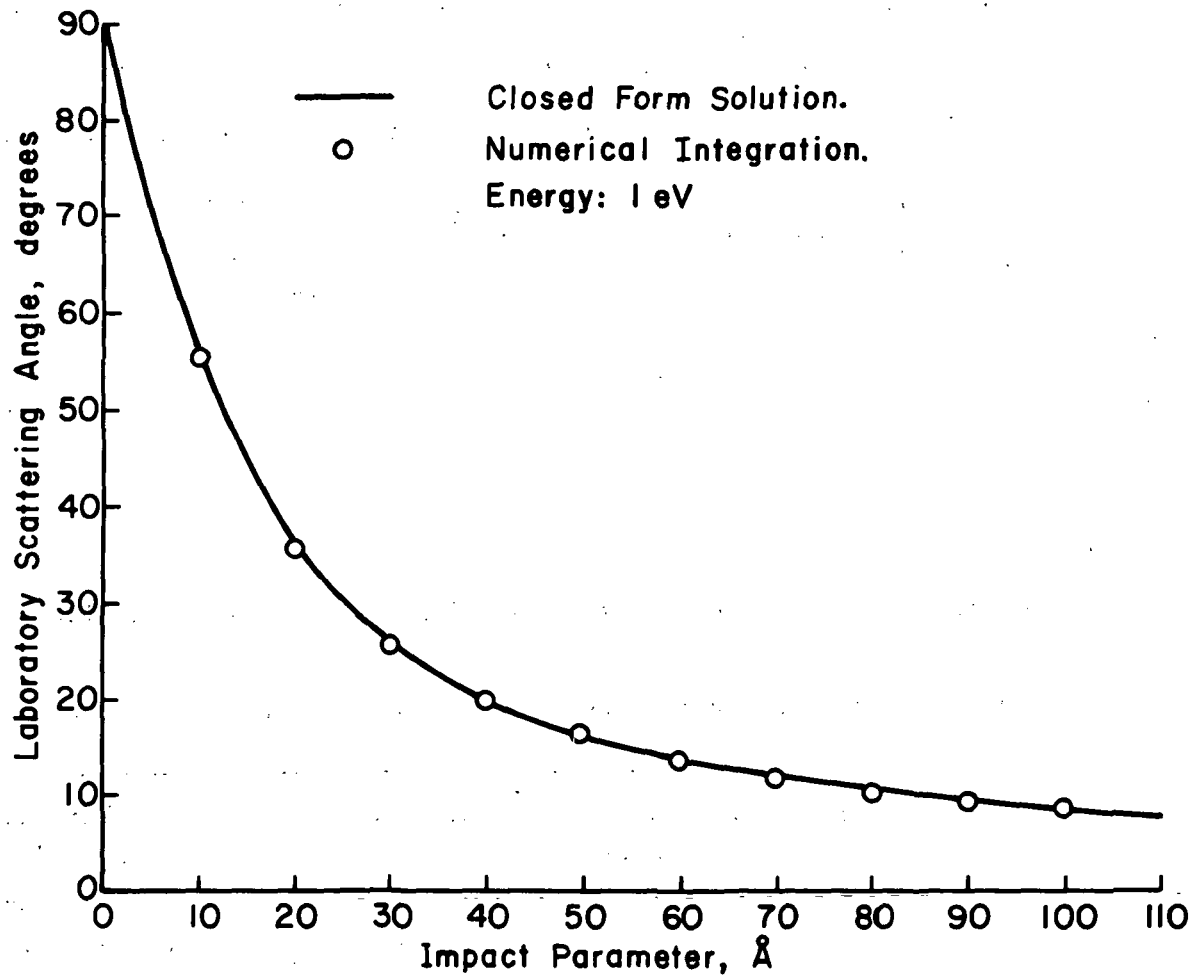


Figure 5-4. Rutherford Scattering: Comparison of closed form and numerical integration for $\text{Ar}^+ \rightarrow \text{Ar}^+$ @ 1 eV.

Argon Interaction Potential

To use a classical approximation for argon-argon scattering it is necessary to obtain a potential function describing the argon-argon pair interaction. Barker, Fisher and Watts¹¹ have developed an argon pair potential that is recognized as being in best overall agreement with experimental data from a number of sources such as specific heats, internal energies, viscosities, solid state parameters, and scattering data. Their expression for the argon isolated pair potential is the Barker-Pompe form:

$$\phi(r) = \epsilon \left[e^{-\alpha(R-1)} \sum_{i=0}^5 A_i (R-1)^i - \frac{C_6}{(R^6 + \delta)} - \frac{C_8}{(R^8 + \delta)} - \frac{C_{10}}{(R^{10} + \delta)} \right] \quad (5-13)$$

with $R = r/r_m$.

The Barker-Fisher-Watts coefficients for argon are given in Table 5-3.

Table 5-3. Barker-Fisher-Watts coefficients for the argon pair potential.

<u>Coefficient</u>	<u>Value</u>	<u>Coefficient</u>	<u>Value</u>
ϵ	0.0122448 eV	C_6	1.10727
r_m	3.7612 Å	C_8	0.16971325
A_0	0.27783	C_{10}	0.013611
A_1	-4.50431	α	12.5
A_2	-8.331215	δ	0.01
A_3	-25.2696		
A_4	-102.0195		
A_5	-113.25		

Figure 5-5 is a plot of the pair potential on a linear scale showing

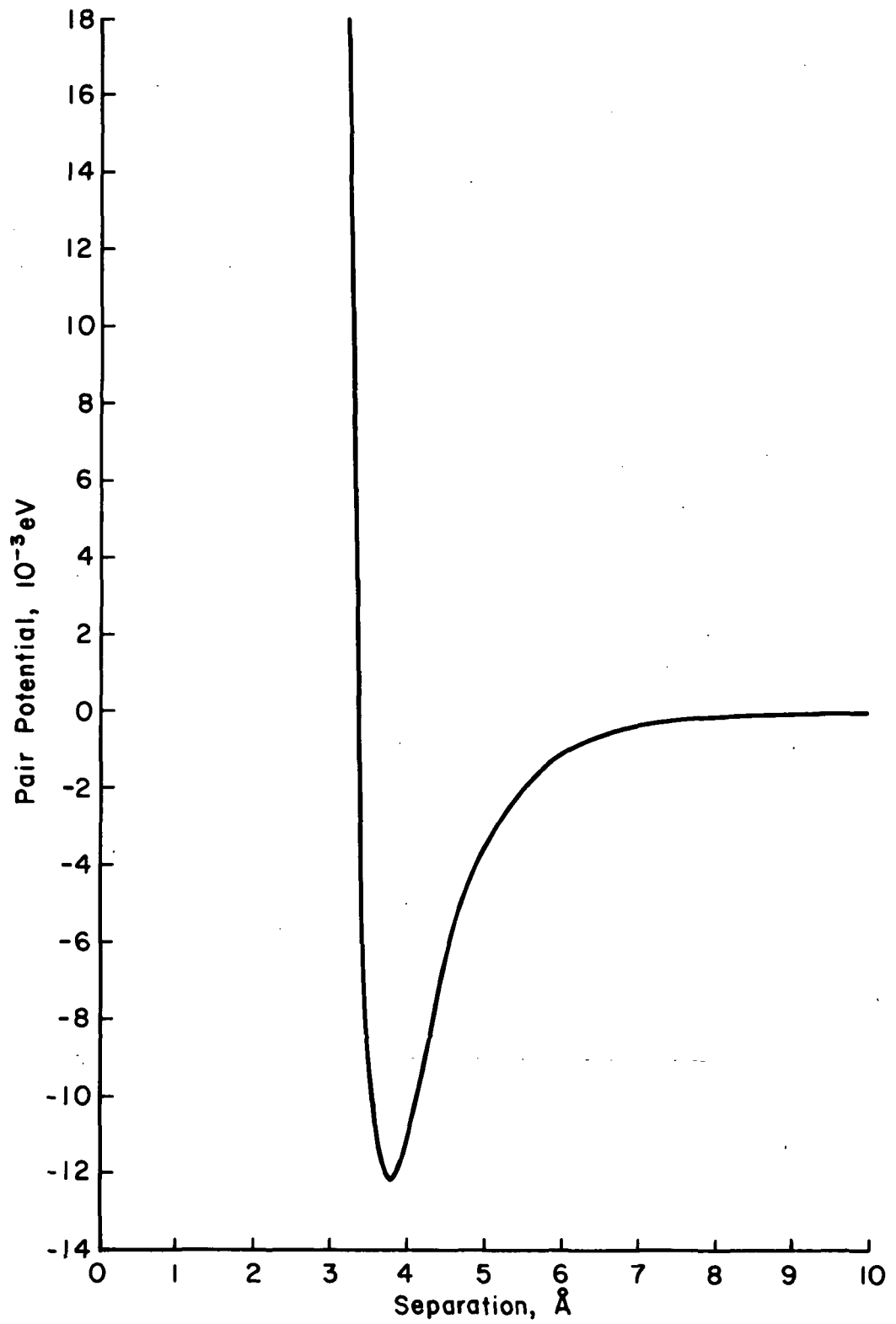


Figure 5-5. Barker-Fisher-Watts argon pair potential: attractive well and zero crossing.

the attractive part of the potential, the minimum, and the zero crossing. The depth of the attractive well is given by the coefficient $\epsilon = 0.0122448$ eV. This is an energy four or five orders of magnitude lower than most beam energies of interest. Thus, in scattering, attractive forces will be of little consequence compared to the dominant repulsive core potential.

Figure 5-6 shows the argon pair potential on a logarithmic energy scale for the repulsive core region.

Calculated Results

Table 5-4 contains some of the laboratory scattering angles calculated as a function of impact parameter and incident energy using the Barker-Fisher-Watts argon potential.

Table 5-4. Calculated laboratory scattering angles as a function of incident energy and impact parameter for argon.

Impact Parameter, Å	Energy of incident atom, eV				
	1.0	10	100	500	1000
0.0	90.0	90.0	90.0	90.0	90.0
0.2	84.9	82.5	80.1	77.5	75.8
0.4	79.9	75.0	70.2	65.0	61.7
0.6	74.8	67.4	60.2	52.5	47.9
0.8	69.7	59.9	50.0	40.2	34.6
1.0	64.6	52.3	39.7	28.2	22.4
1.2	59.4	44.9	29.5	17.4	12.4
1.4	54.2	37.8	19.9	9.02	5.59
1.6	49.0	31.1	12.0	4.00	2.22
1.8	43.7	25.0	6.93	1.81	0.94
2.0	38.3	19.4	4.16	0.95	0.49
2.2	32.8	14.6	2.60	0.57	0.29
2.4	27.3	10.3	1.60	0.34	0.17
2.6	21.8	6.78	0.93	0.19	0.10
2.8	16.3	4.03	0.49	0.10	0.05
3.0	11.0	2.10	0.24	0.05	0.02
3.2	6.28	0.90	0.10	0.02	0.01
3.4	2.49	0.26	0.03	0.01	
3.6	0.01		0.01		

Figure 5-7 is a plot of some of the numerically integrated scattering angles given in Table 5-4. These curves show the basic trends of the

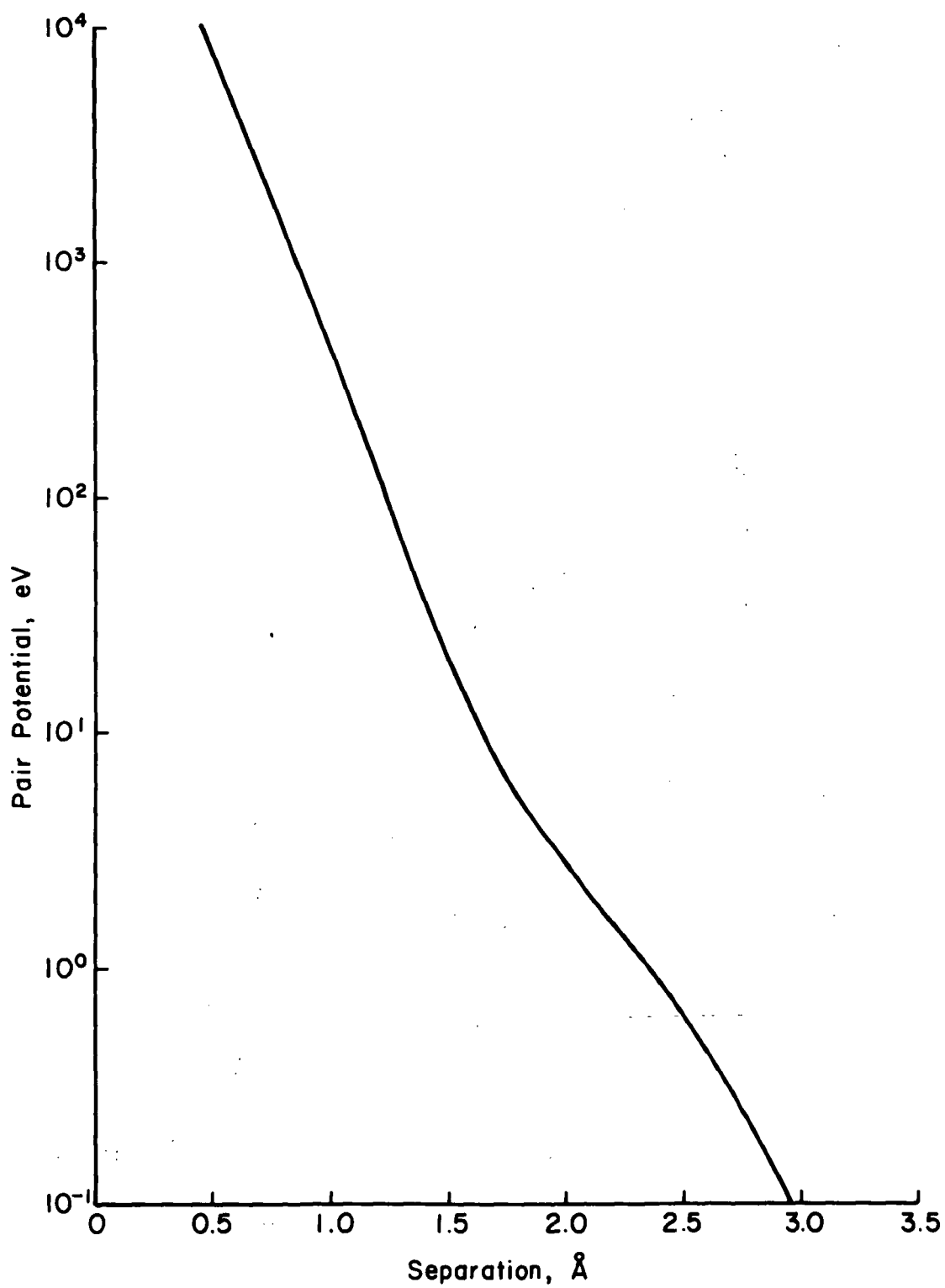


Figure 5-6. Argon pair potential repulsive core.

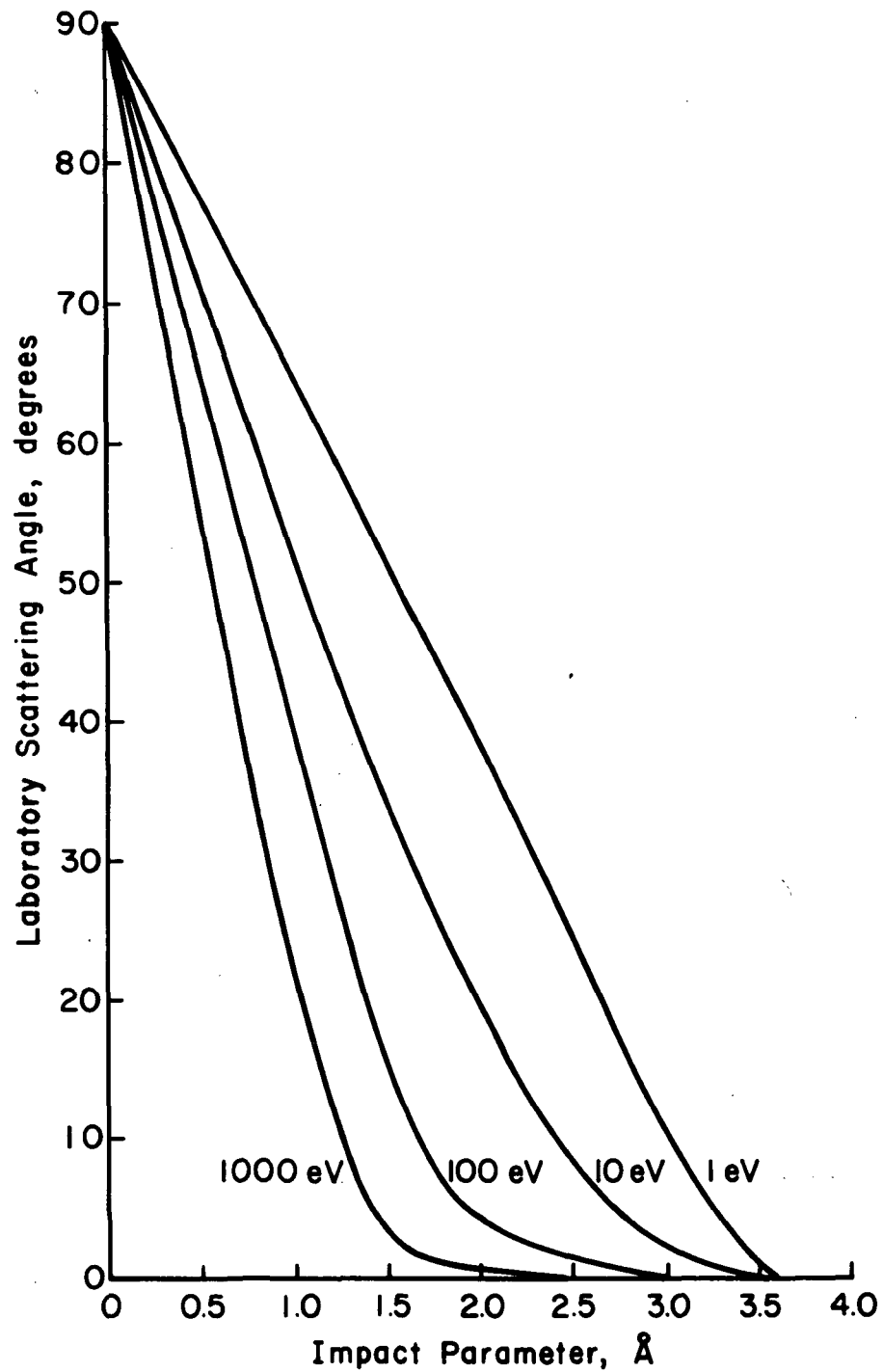


Figure 5-7. Calculated argon laboratory scattering angles as a function of impact parameter at various energies.

scattering in this energy range. Lower energy atoms are scattered at larger angles for the same impact parameter implying an increase in calculated cross sections as the beam energy is reduced.

Although, using classical calculations, it is not possible to obtain correct values for the total cross sections,⁹ it is possible to correlate cross sections for scattering through angles greater than some minimum angle θ_0 . The classical cross section is taken as the area of a circle of radius equal to the impact parameter for this minimum angle.

Table 5-5 gives representative calculated values for the total cross section, $Q(\theta > 10^\circ)$, for scattering through angles greater than 10° as a function of energy.

Table 5-5. Total cross section for argon scattering through angles greater than 10° .

E eV	Q ($\theta > 10^\circ$) $\text{\AA}^2$
1	29.1
3	24.2
10	18.4
30	12.9
100	8.76
300	6.71
1000	5.00
2000	4.14

For small angle scattering, an approximate correlation is achieved for potentials that approach exponential behavior in the energy range of interest. The Barker-Fisher-Watts potential exhibits exponential behavior over certain energy ranges at small separations. This correlation for the cross section as a function of energy can be seen by plotting

the square root of the cross section versus the logarithm of the fourth root of the cross section divided by the energy.¹² Figure 5-8 is a plot of the correlated parameters showing the behavior over approximately four decades for $Q(\theta > 10^\circ)$. The correlation appears to be nearly linear in two separate energy ranges, $E > 100\text{eV}$ and $E < 100\text{eV}$. Such a correlation can be used for interpolation and for minor extrapolation.

Potential for Other Rare Gases

Barker et. al.¹³ have developed potentials for two other rare gases, krypton and xenon, using nearly the same analytical form for the potential as for argon with similar adjustable parameters. The form for these potentials is:

$$\phi(r) = \epsilon \left[e^{\alpha(1-R)} \sum_{i=0}^5 A_i (R-1)^i - \frac{C_6}{(R^6 + \delta)} - \frac{C_8}{(R^8 + \delta)} - \frac{C_{10}}{(R^{10} + \delta)} \right] + \epsilon \left[P(R-1)^4 + Q(R-1)^5 \right] e^{\alpha(1-R)},$$

for $R > 1$ (5-14)

$$\phi(r) = \epsilon \left[e^{\alpha(1-R)} \sum_{i=0}^5 A_i (R-1)^i - \frac{C_6}{(R^6 + \delta)} - \frac{C_8}{(R^8 + \delta)} - \frac{C_{10}}{(R^{10} + \delta)} \right], \text{ for } R < 1$$

(5-15)

with $R = r/r_m$. The coefficients for krypton and xenon are given in Table 5-6.

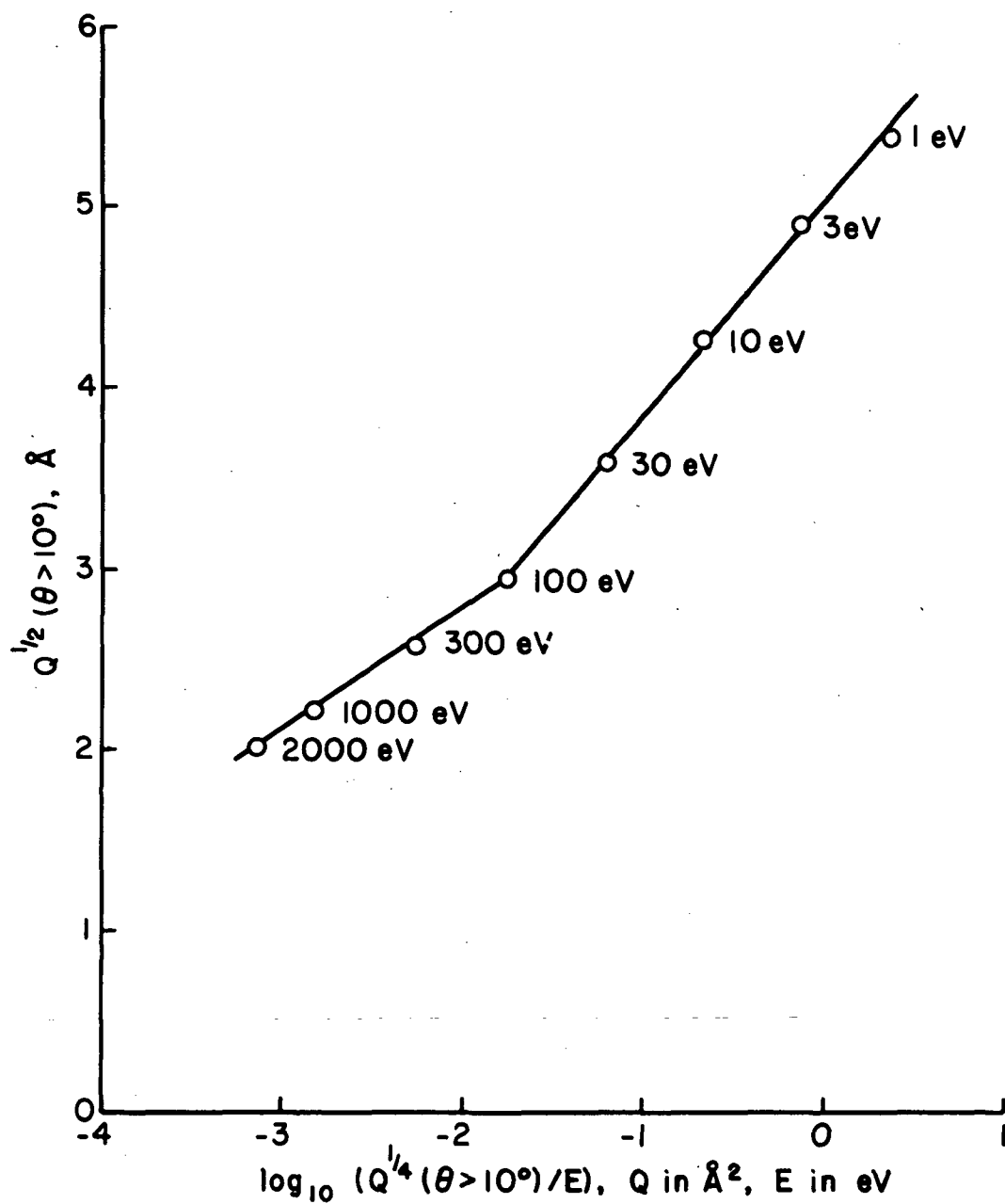


Figure 5-8. Correlation parameters for cross section and energy for argon scattering through angles greater than 10° .

Table 5-6. Coefficients for krypton and xenon pair potentials (5-14) and (5-15).

<u>Coefficient</u>	<u>Kr Value</u>	<u>Xe Value</u>
ϵ	0.01740 eV	0.02421 eV
r_m	4.0067 Å	4.3623 Å
A_0	0.23526	0.2402
A_1	-4.78686	-4.8169
A_2	-9.2	-10.9
A_3	-8.0	-25.0
A_4	-30.0	-50.7
A_5	-205.8	-200.0
C_6	1.0632	1.0544
C_8	0.1701	0.1660
C_{10}	0.0143	0.0323
α	12.5	12.5
δ	0.01	0.01
P	-9.0	59.3
Q	68.67	71.1

Figure 5-9 compares the pair potentials for Ar, Kr and Xe showing the increase in effective radius of the atom as the atomic number increases. The general character of the like-particle scattering from Kr and Xe in the classical approximation would be similar to the Ar scattering since the shapes of the potentials are very nearly the same as the Ar potential. However, the corresponding cross sections for the heavier gases should be larger in the energy range displayed in Fig. 5-9.

Calculated Differential Cross Sections for Argon

The necessity for calculating differential cross sections directly from a potential might reasonably be questioned; however, Massey and Gilbody have this to say:

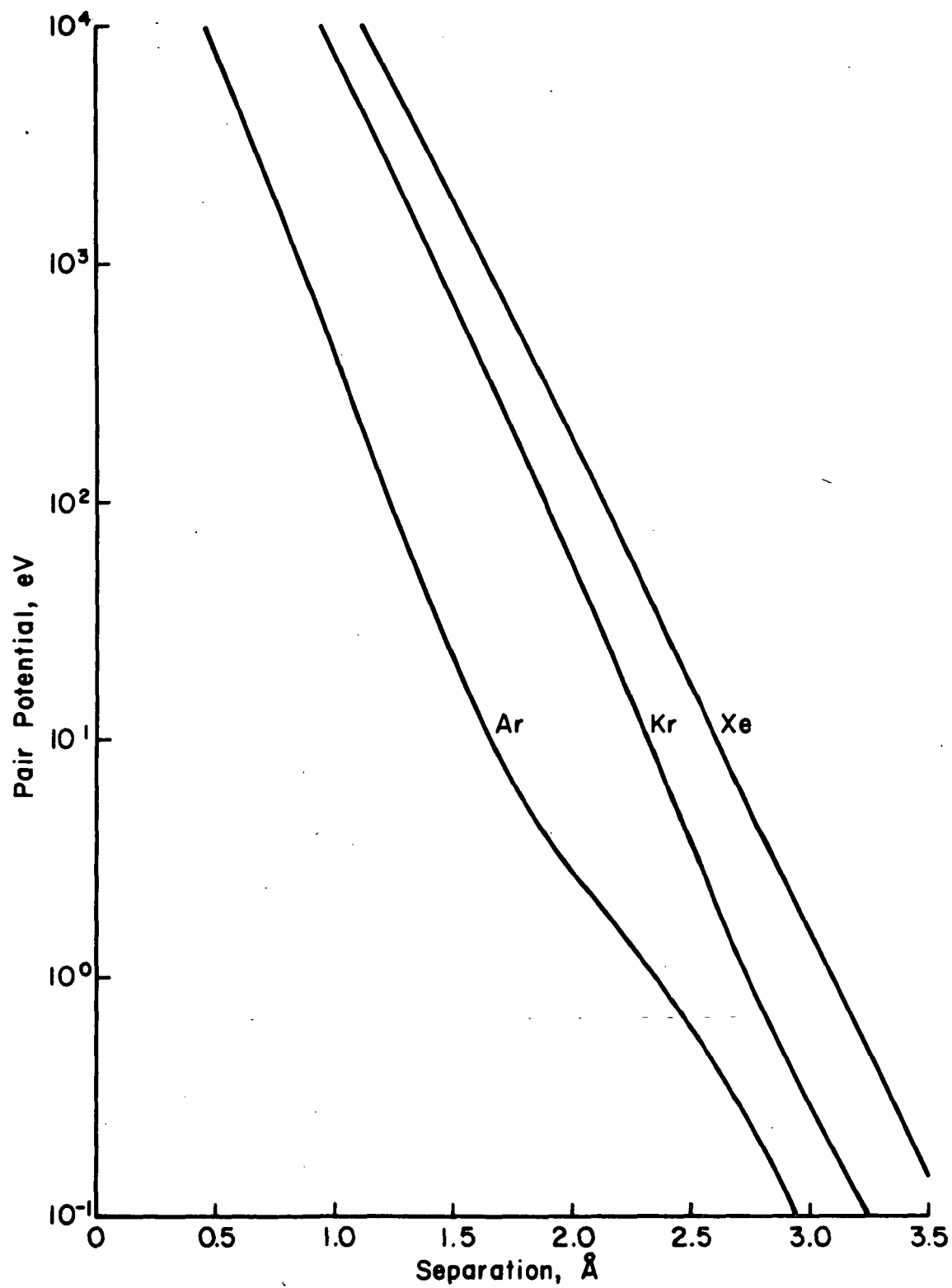


Figure 5-9. Comparison of pair potentials for rare gases, Ar, Kr, Xe.

"Up to the time of this writing (1974) measurements of differential cross-sections for elastic scattering have been almost entirely confined to beams of positive ions in gases. For neutral atom beams observations have been confined to the 'total' scattering outside a particular mean scattering angle in the laboratory system. Measurements of this kind have also been carried out for ion beams."¹⁴

The thorough search conducted by Massey and Gilbody thus uncovered no data relating to the important neutral-neutral momentum loss process.

To determine the average momentum loss of argon atoms that suffer collisions with other argon atoms, the differential cross section must first be calculated. The differential cross section is given by⁹

$$\sigma(\theta) = - \frac{s}{\sin\theta} \frac{ds}{d\theta}, \quad (5-16)$$

where s is the impact parameter and $\sigma(\theta)$ is the differential cross section measured in $\text{m}^2/\text{steradian}$. Data of the kind given in Fig. 5-7 were used to calculate the differential cross section using Eq. (5-16). As an example, the differential scattering cross section for 500eV argon is plotted in Fig. 5-10. It can be seen from this curve that most of the scattering occurs at angles that do not contribute significantly to momentum loss; also, the differential cross section tends to zero as the scattering angles approach 90° , which is the value for maximum momentum loss.

Ion-neutral Collisions

A potential function was not available describing the interaction between an argon ion and a neutral argon atom. However, because only

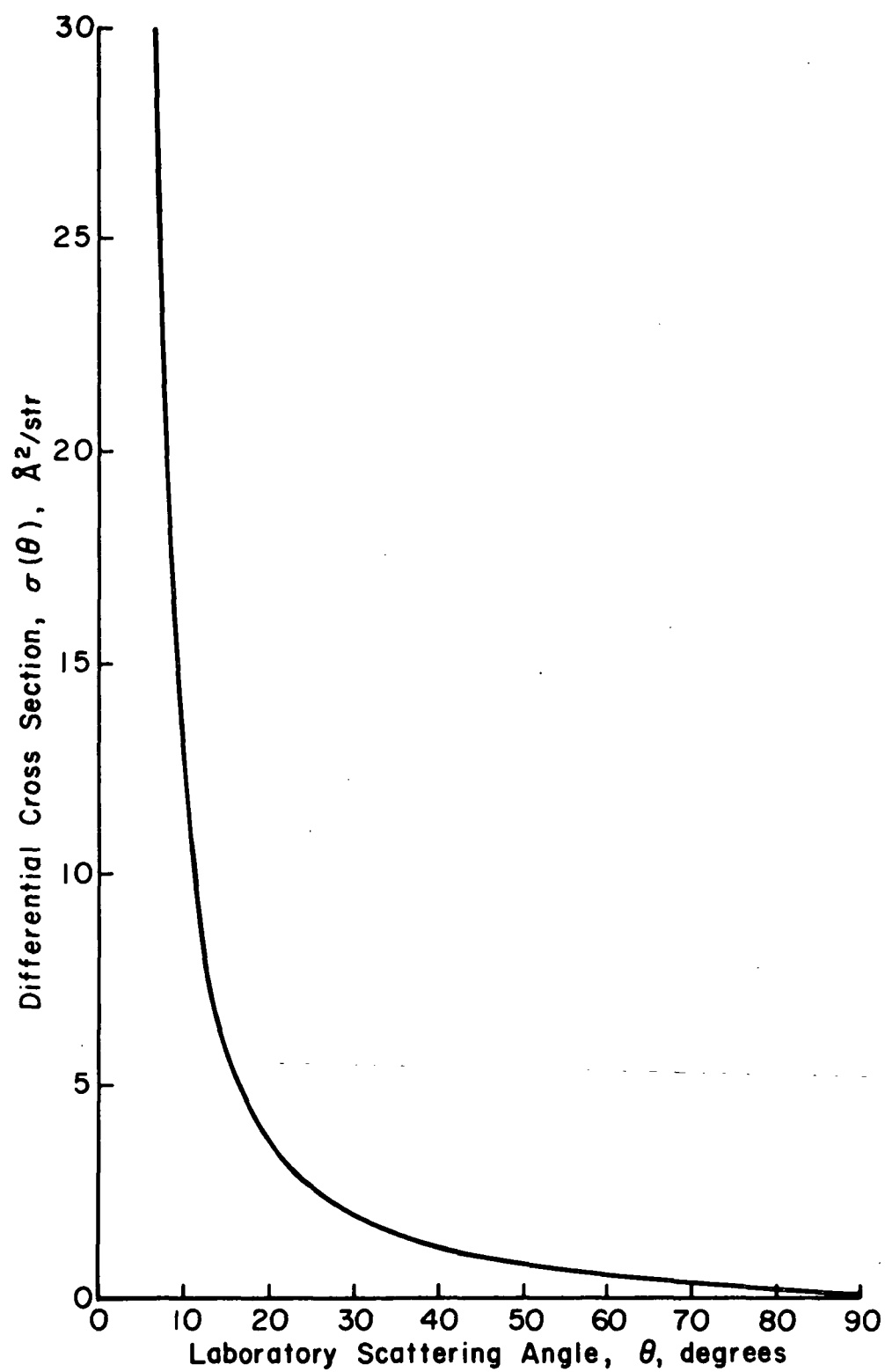


Figure 5-10. Calculated differential elastic scattering cross-section for 500eV Ar-Ar collisions.

one of the particles is charged, the interaction at large distances can be of no lower order than dipole-monopole which would imply a modification in the potential to include attractive r^{-4} and higher-order potential terms. The repulsive core potential would be altered by the absence of one electron compared to the Ar-Ar interaction; but, generally, the ion-atom interaction should remain of similar order of magnitude to the atom-atom interaction at energies above several eV. The absence of one electron in the ion-atom interaction may tend to weaken the repulsion, thereby reducing the differential scattering cross section compared to the argon-argon differential cross section.

Some experimental data are available for Ar^+ -Ar collisions at 400eV.¹⁵ These experimental data, expressed as a differential cross section, are compared in Fig. 5-11 to the calculated differential cross section for 500eV Ar-Ar collisions.

As expected, the Ar^+ -Ar differential cross section is less than that for Ar-Ar; however, the general trends of the data are similar in each case. The experimental data reported by Aberth and Lorents¹⁵ oscillates somewhat but the linear fit shown in Fig. 5-11 is a fair representation of the magnitudes and trends in the data.

Even when the interaction cross sections are directly compared between Ar^+ -Ar and Ar-Ar collisions, the neutral-neutral collisions remain the dominant process to be considered for momentum and energy loss in the beam.

Forward Momentum Loss in Beam

In a collision in which an argon atom is scattered through an angle θ , it will have lost a fraction of its forward momentum equal to $(1 - \cos^2 \theta)$. The average fraction of forward momentum loss, f , for scattered argon

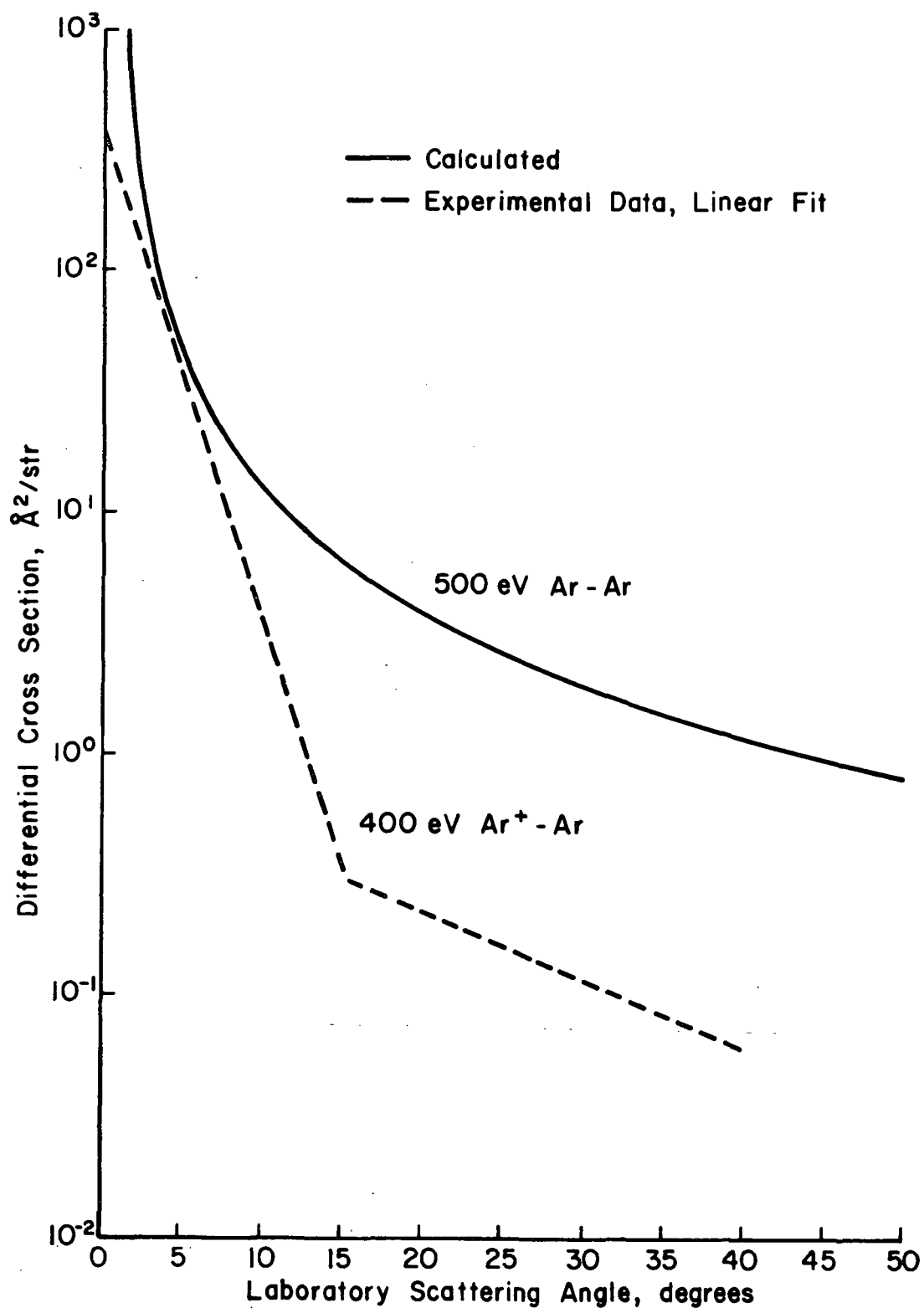


Figure 5-11. Comparison of differential scattering cross sections for Ar-Ar (calculated) and Ar^+ -Ar (experimental).

atoms is:

$$f = \frac{\int_0^{\pi/2} (1-\cos^2\theta) \sigma(\theta) 2\pi\sin\theta d\theta}{\int_0^{\pi/2} \sigma(\theta) 2\pi\sin\theta d\theta} \quad (5-17)$$

This fraction can be thought of as the ratio of two cross sections: i.e. the ratio of a momentum loss cross section to the total scattering cross section. The fractional loss of momentum P from a beam traversing a distance dx through a medium containing a number density n of scattering centers of total scattering cross section Q is:

$$\frac{\Delta P}{P} = f n Q dx \quad (5-18)$$

where f is the average fractional loss per collision given in Eq. (5-17). The product fQ can be considered as a single parameter or cross section, Q_p , related to the rate of momentum decay in the beam. Equation (5-19) is the customary form for the widely used defined cross section for viscosity.¹⁶

$$Q_p = \int_0^{\pi/2} (1-\cos^2\theta) \sigma(\theta) 2\pi\sin\theta d\theta \quad (5-19)$$

Using differential cross sections calculated for a number of incident energies, the integrals of the form given in Eq. (5-19) were evaluated to yield cross sections for directed momentum loss as a function of beam energy. Figure 5-12 displays the results of these calculations. The cross section decreases rapidly as energy increases for the lower energy; however, between 500eV and 1000eV where a large fraction of sputtering is done, the cross section changes more slowly.

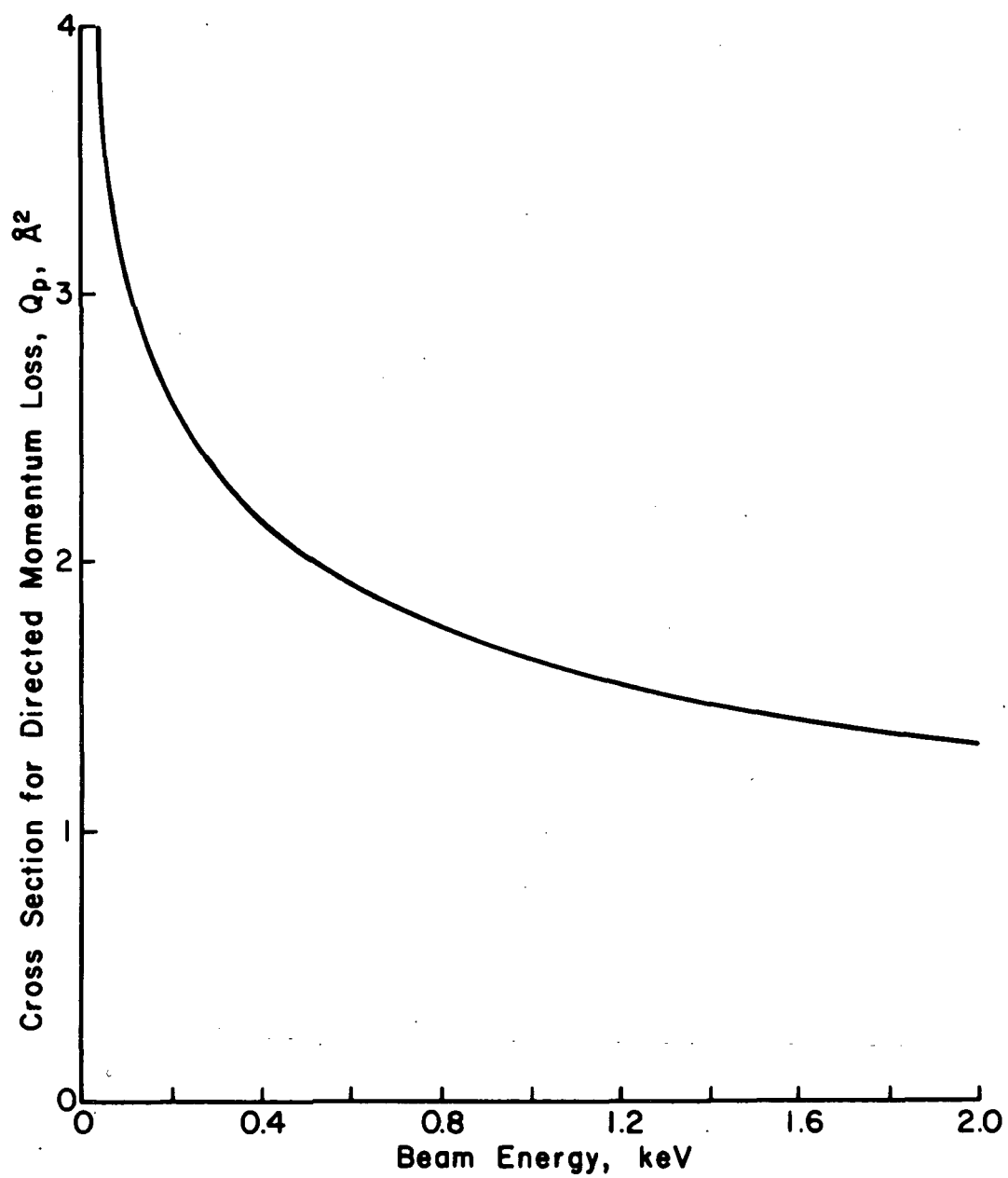


Figure 5-12. Directed momentum loss cross section for Ar-Ar collisions as a function of beam energy.

The rapid decrease in the directed momentum loss cross section with increasing energy is qualitatively similar to the behavior of the total cross section for scattering through angles greater than 10° (Table 5-5). Figure 5-13 displays a correlation in the directed momentum loss cross section similar to the correlation of Fig. 5-8; again, two distinct linear segments are observed that can facilitate interpolation or extrapolation of the data.

Figure 5-14 is a plot of the mean free path for directed momentum loss times the pressure versus beam energy. At a given energy, dividing the ordinate by the background pressure yields a measure of the mean free path for directed momentum loss in the beam. The curve was calculated from the data of Fig. 5-12 assuming ideal gas behavior and a temperature of approximately 300°K.

The anticipated use of Fig. 5-14 is in the determination of acceptable background pressures for ion beam etching. The directed momentum loss cross section is identical with the non-directed energy loss cross section. An energy loss in the beam of up to 10 percent might be expected to be acceptable. The source-to-substrate distance should therefore correspond to about $1/10$ of a mean free path. At 1000eV, for example, the pressure times distance should equal about 25×10^{-3} Torr-cm, or less, for a 10 percent, or less, loss in beam energy. This calculation, then, permits a tradeoff to be made between pumping capacity and the ion beam energy and momentum losses. The simple method described should be adequate for losses up to 10 or 20 percent of the initial beam energy, but larger losses will probably require a more detailed calculation that will take into account both the mean energy and the distribution of particle energies as a

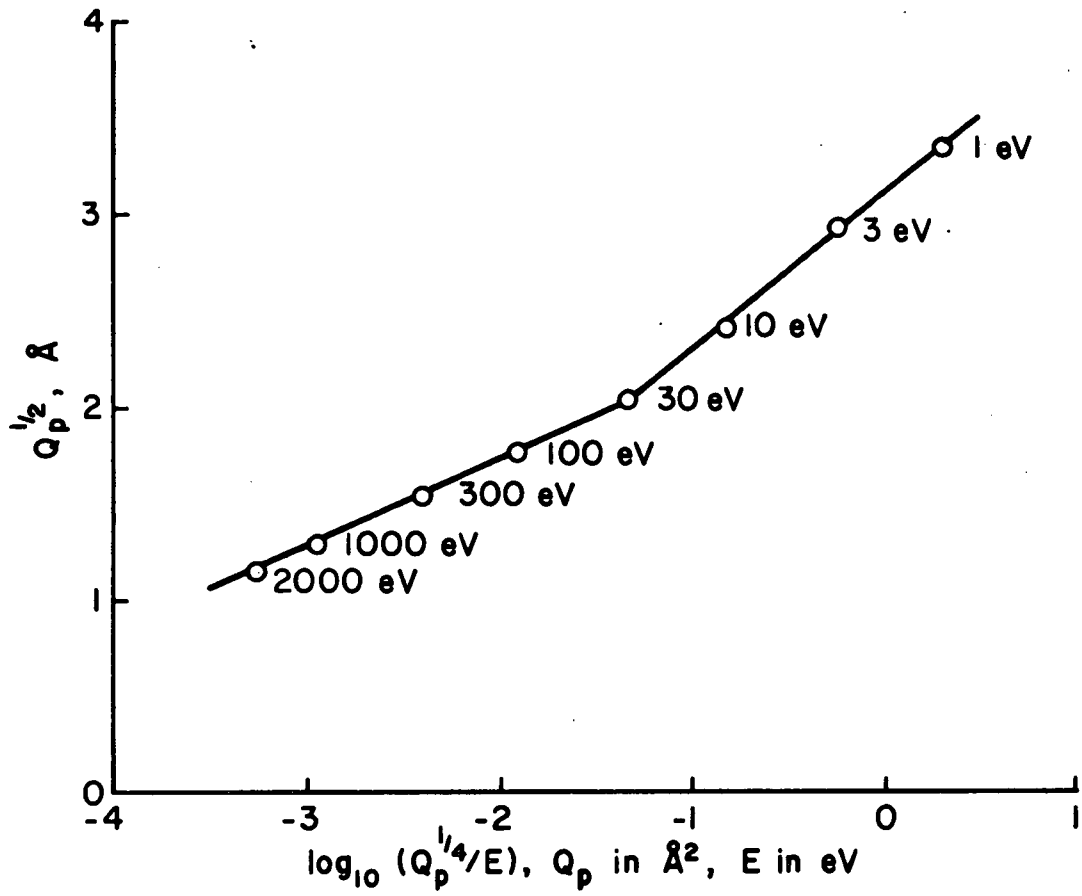


Figure 5-13. Directed momentum loss cross section correlated with beam energy.

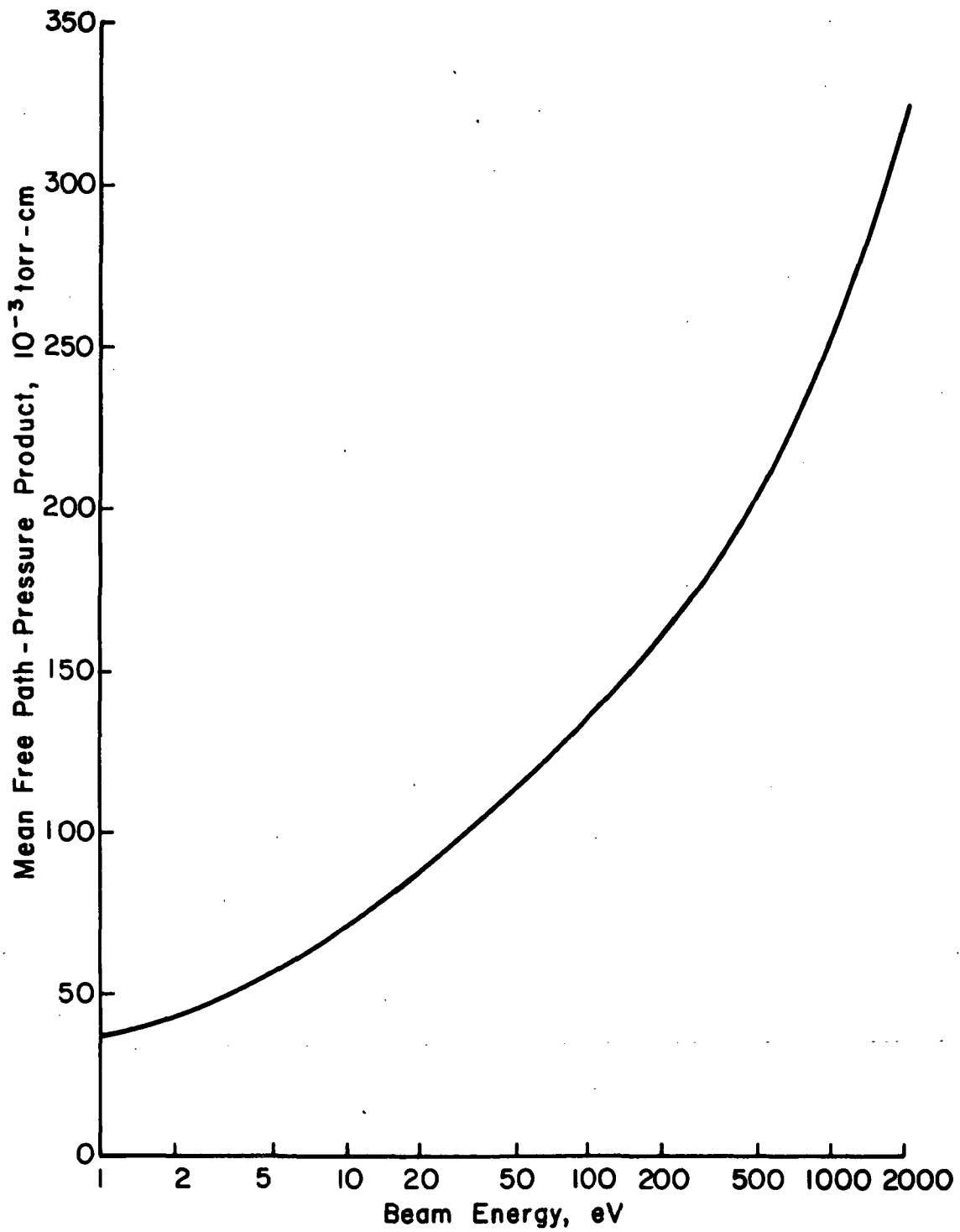


Figure 5-14. Argon directed momentum loss: mean free path - pressure product as a function of beam energy.

function of distance. Knock-on atoms should also be included as well as beam atoms in a more detailed analysis.

In comparison to this 25×10^{-3} Torr-cm for a 10 percent energy loss in the beam, a 10 percent charge exchange requires only 1.4×10^{-3} Torr-cm. It should be clear that a substantial amount of charge exchange can take place without significantly affecting the momentum, energy, or, to the first approximation, the sputtering capability of the beam.

The interaction of a beam of argon ions passing through a background gas of argon is of interest for industrial application of broad-beam ion sources, particularly for etching. The momentum or energy losses in the beam can be used to determine pressure, hence pumping, requirements in etching applications. Because the necessary elastic cross sections for argon-argon collisions have not been available prior to this publication, the calculation of momentum and energy losses in the beam has not previously been possible.

VI. ESTIMATED COSTS OF ION-BEAM SPUTTERING PROCESSES

The introduction of ion-beam sputtering processes into production requires estimates of the costs involved. A costing model, together with preliminary cost estimates, is presented in this section. The assumed costing model is one in which an ion-beam machine is purchased for a specific production etching process, then used for this process at a significant fraction of maximum capacity over the lifetime of the machine. This approach results in the setup and fixturing for the specific etching process being a negligible part of total costs.*

A preliminary analysis indicated that labor, machine, and overhead were the major costs. Material costs could be important in the final product, but were not significant in the incremental process costs. The assignment of overhead costs, if realistic costs are to be obtained, should reflect the manner in which these costs are incurred. Inasmuch as both labor and machine costs can dominate different aspects of sputtering processes, overhead was assigned to both labor and machine costs.

The ion-beam machines included in this study were the complete lines of two different manufacturers. The nominal beam diameters of these machines ranged from 2.5 to 30 cm. A more meaningful measure of machine size, though, is the ion-beam working area. This area was defined as that over which the ion current density varies by ± 5 percent, or less. The substrates to be etched were assumed to completely fill

*In contrast, the typical use of an ion-beam machine in a research laboratory is on a wide range of sputter etching or deposition processes, so that setup and fixturing can easily dominate the costing.

this working area. A more typical case would be circular substrates with clamps and other unused space amounting to about half the working area. The cost per unit area etched would then be about twice that given on the basis of total working area. In any event, it is necessary for the prospective user to evaluate the use efficiency for the working area, and correspondingly adjust the costs presented herein.

To facilitate comparison, all machines were assumed to be operated with 500 eV argon ions. The use of argon is, of course, almost universal in ion-beam machines. The 500 eV energy was selected as a compromise between low energy operation to minimize substrate damage and high energy operation to maximize material removal. In practice, higher ion energies would usually result in a beam current increases of less than 50 percent due to power supply limitations. Decreased ion energies, though, would typically reduce beam current according to Child's law ($J \propto (eV)^{3/2}$).

The ion etch dose is given in mA-hr/cm² to avoid the assumption of specific materials. A specific application will therefore require a specific sputtering yield to translate the etch dose in mA-hr/cm² into an etch depth. To give some indication of the magnitude of an etch dose, the typical etch of a solid-state electronics circuit would be a small fraction of one mA-hr/cm², while a deep surface texturing could easily require a few mA-hr/cm².

To help convert etch doses in mA-hr/cm² to etch depths, Table 6-1 is included. The etching rates included therein are in the usual application units of angstroms/min for an ion current density of 1 mA/cm², with argon ions at 500 eV the bombarding species. Most etching rates were calculated from sputtering yields (atoms or molecules per incident ion)

TABLE 6-1. ETCH RATES FOR 500 EV ARGON IONS AT 1 MA/CM².

Target	Å/min.	Ref.	Target	Å/min.	Ref.
Be	190	1	Gd	1000	2
C	40	2	Dy	1000	2
Al	630	1	Er	870	2
Si	320	1	Hf	600	1
Ti	340	1	Ta	380	1
V	340	1	W	340	1
Cr	540	1	Re	460	1
Fe	500	1	Os	440	1
Co	500	1	Ir	540	1
Ni	560	1	Pt	780	1
Cu	870	1	Au	1500	1
Ge	920	1	Pb	2700	3
Y	860	2	Th	740	1
Zr	570	1	U	650	1
Nb	390	1	CdS (10 $\bar{1}$ 0)	2100	4
Mo	470	1	GaAs (110)	1500	4
Ru	580	1	GaP (111)	1400	4
Rh	650	1	InSb (unknown)	1400	4
Pd	1100	1	LiNbO ₃ (Y-cut)	400	5
Ag	2000	1	PbTe (111)	3400	4
Sn	1500	2	SiC (0001)	320	4
Sm	970	2	SiO ₂ (Y-cut)	400	5

in literature,¹⁻⁴ although a few were obtained directly in angstroms/min.⁵

Machine Costs

The purchase prices of the machines used in this study are shown in Fig. 6-1 as a function of ion-beam working area. These 1977 prices included ion source, electronics console, vacuum facility, and fixturing. (The vacuum facility was about half of the total price for the smallest machines and dropped to about one-fifth of the total price for the largest machines.) These prices were depreciated linearly over an assumed 5-year lifetime. An operating schedule of 1000 hr/yr was assumed to translate the depreciation cost into a cost per hour for the machine.* An overhead assignment was made on the basis of floor area occupied. This floor area consisted of the minimum rectangular floor area of the consoles involved plus a 3-foot wide aisle surrounding this rectangle. The extra area represented by the aisle was felt necessary, not only for maintenance and repair, but also for safety in view of the high voltages involved in the equipment. The assigned overhead was assumed to be 25 dollars/ft²-yr, and was distributed over the 1000 operating hr/yr in the same manner as the depreciation costs. The total machine costs (depreciation plus overhead) are shown in Fig. 6-2 as a function of ion-beam working area. These costs range from about 5 to over 20 dollars/hr. The values shown in Fig. 6-2 are, of course, dependent on the costing assumptions. The assumption most subject to variation is felt to be the overhead assignment per square foot.

*The 1000-hr operating schedule is about half the usual 2080-hr single-shift work year. The reduction from 2080 is assumed to result from startup and shutdown times, required maintenance and the usual work scheduling problems.

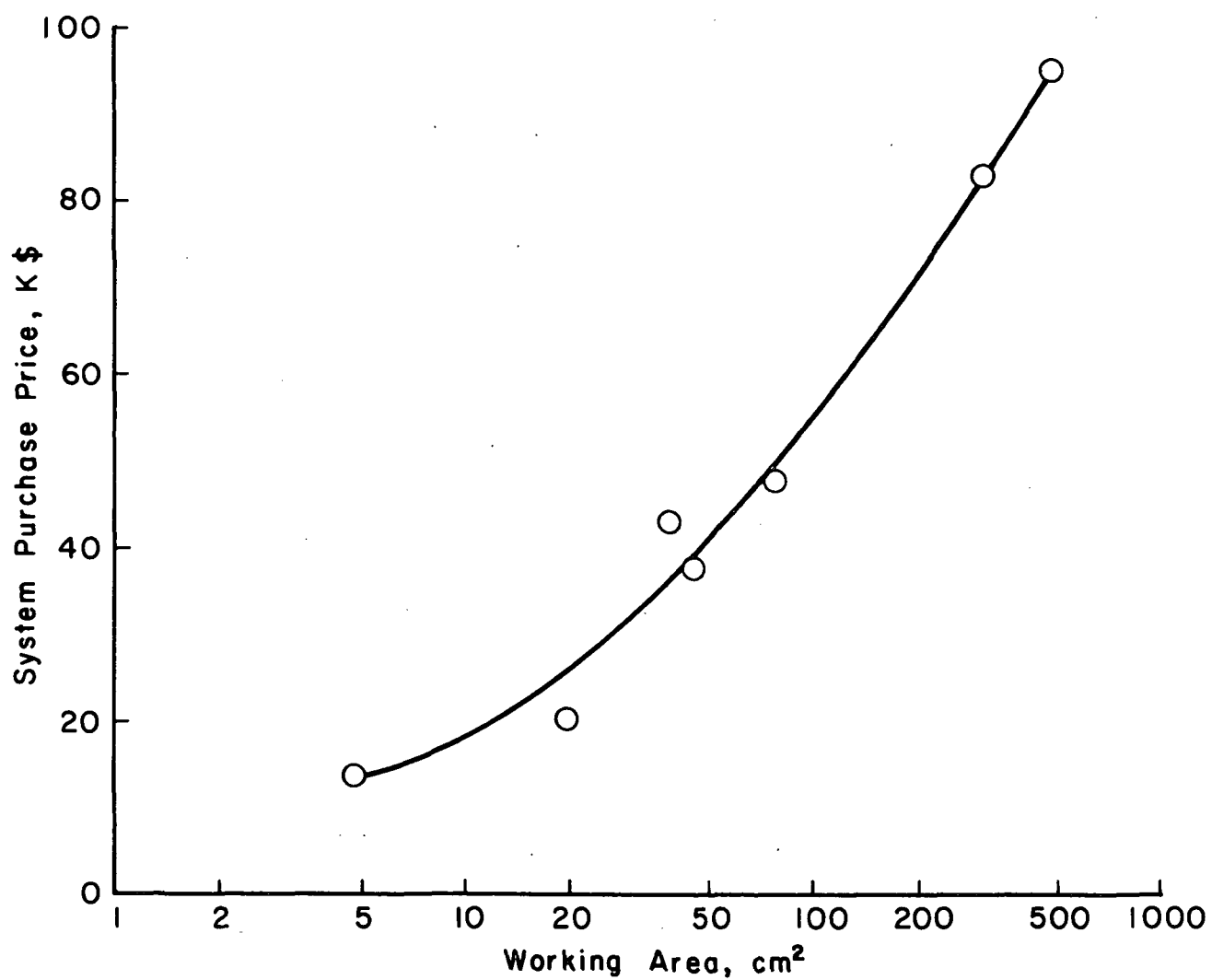


Figure 6-1. Machine purchase price (1977), including ion source, electronics console, vacuum facility, and fixturing.

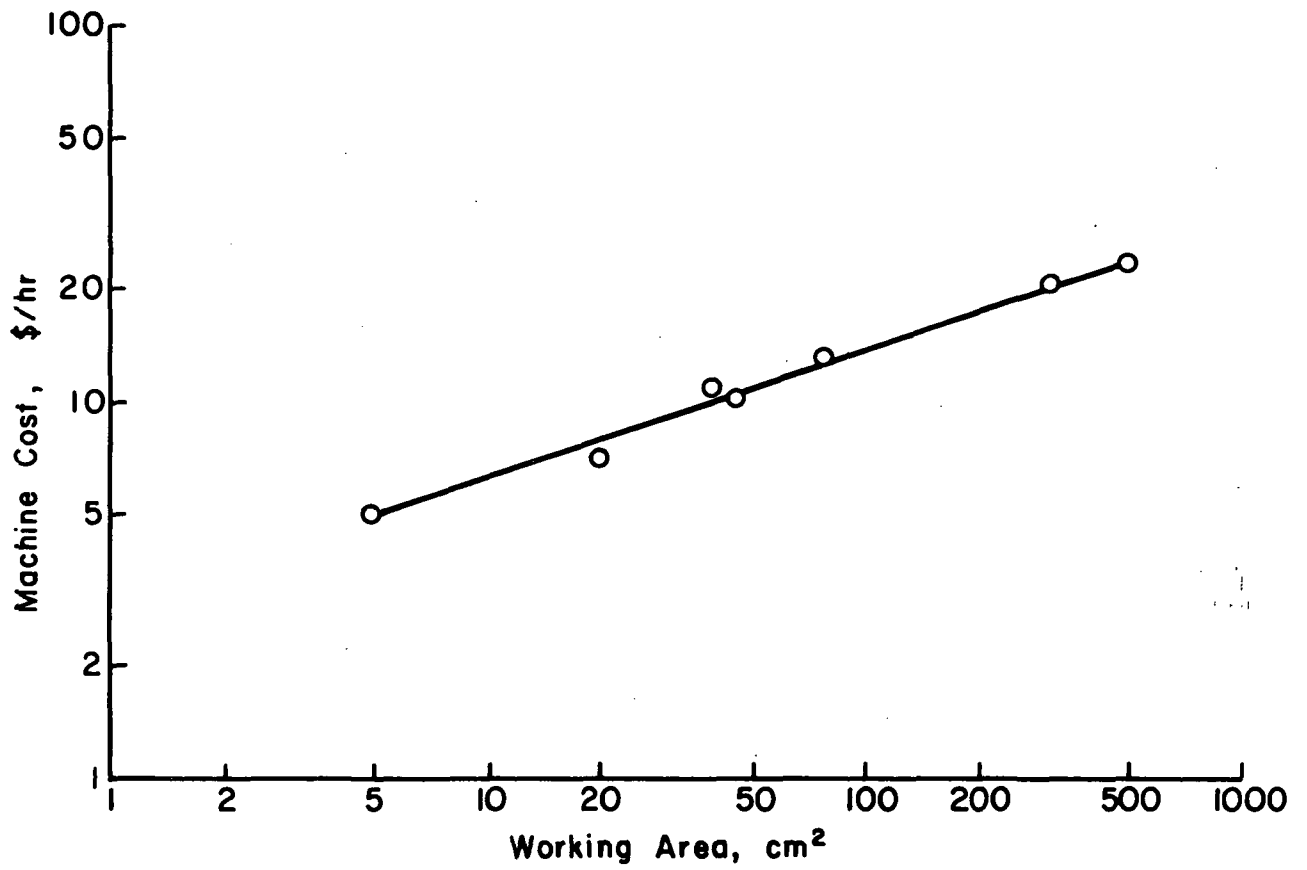


Fig. 6-2. Total machine cost, including both depreciation and overhead, as a function of ion-beam working area.

The fraction of the total machine cost that is in the overhead assignment is shown in Fig. 6-3. The overhead is seen to range from nearly half for small machines to less than one-fifth for large machines. From Fig. 6-3, then, it appears that the total machine costs used herein are not sensitive to the overhead assignment made, at least for the larger machines.

The machine cost per run was assumed to be $\frac{1}{2}$ hour plus whatever time was required for the etch dose used. The $\frac{1}{2}$ hour* includes loading, pump-down, unloading, and ion source maintenance. Some ingenuity is assumed for reaching this $\frac{1}{2}$ hour time, but not more than that which would normally be expected as the result of some trial operations. For example, if the substrate fixture must cool before the substrates can be removed from the fixture, then a duplicate fixture should be provided so that the cooling can take place while a new load is installed and etched.

Labor Costs

A technician level of labor was assumed with a direct cost of 10 dollars/hr. A labor overhead of 150 percent was also assumed, giving a total labor cost of 25 dollars/hr. The required labor for a run was assumed to consist of a fixed time of $\frac{1}{2}$ hour, plus a time dependent on the substrate area being etched, plus another time dependent on the etch dose. The area dependent time was equal in hours to the working area in

*Exposure of the ion source when it is too hot will result in oxide formation on the stainless steel surfaces that will change the source operating characteristics. To achieve the $\frac{1}{2}$ hour time assumed here will probably require either an additional vacuum valve so that the ion source can remain at vacuum or the use of some rapid cooldown technique such as cool nitrogen gas in the bell jar.

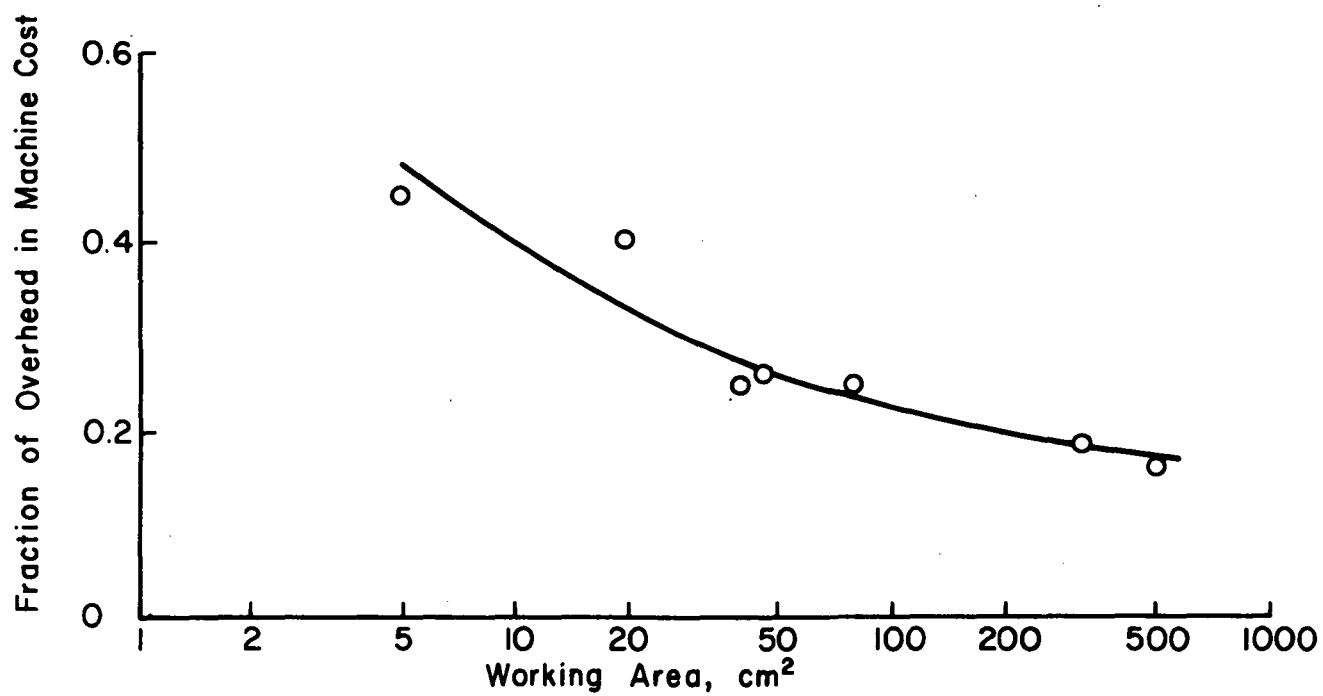


Fig. 6-3. Fraction of total machine cost that is due to overhead, as a function of ion-beam working area.

cm^2 divided by 1000. The etch-dose dependent term was equal in hours to the etch dose in mA-hr/cm^2 divided by 10.

The $\frac{1}{2}$ hour of labor is essentially the same $\frac{1}{2}$ hour assumed for machine time. The additional times are associated with loading and unloading substrate fixtures as well as cleaning extraneous sputtered material from various surfaces. Proper fixture and vacuum chamber design will permit most of these additional tasks to take place outside of the machines. Note that the total labor time per run is not affected strongly by the length of the run. This is in accord with observation in that much of the cleaning and maintenance is associated with the exposure to atmosphere.

Etch Costs

Using the costing model described above, the fixed and variable etch costs were calculated and presented in Figs. 6-4 and 6-5. The fixed cost is essentially the cost for a very small etch dose. The variable cost is the incremental cost for each additional etch dose of 1 mA-hr/cm^2 . The fraction labor cost in the fixed and variable costs is shown in Figs. 6-6 and 6-7.

It is clear that the fixed cost is labor intensive and therefore very sensitive to the loading, cleaning, and maintenance procedures used. The high fraction of labor in the fixed cost is probably also responsible for the small scatter in Fig. 6-4. That is, the scatter should be expected to be small if labor is the major cost contribution and consistent rules are used for calculating the required labor.

In contrast, the variable etch cost is machine intensive, and thus sensitive to machine performance and cost. There is in Fig. 6-5, for

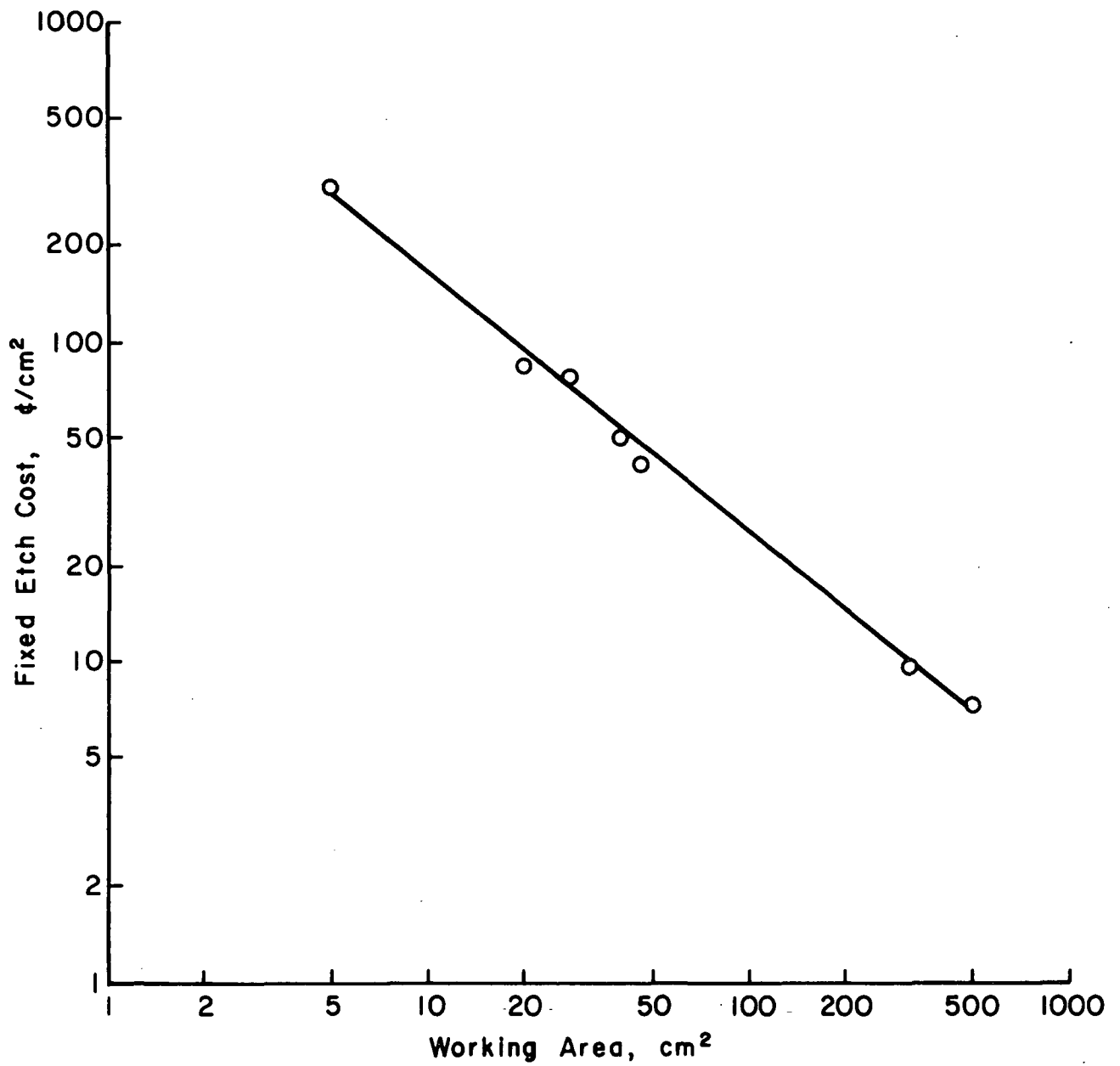


Fig. 6-4. Fixed etch cost as a function of ion-beam working area. The fixed etch cost is essentially the cost for a very small etch dose.

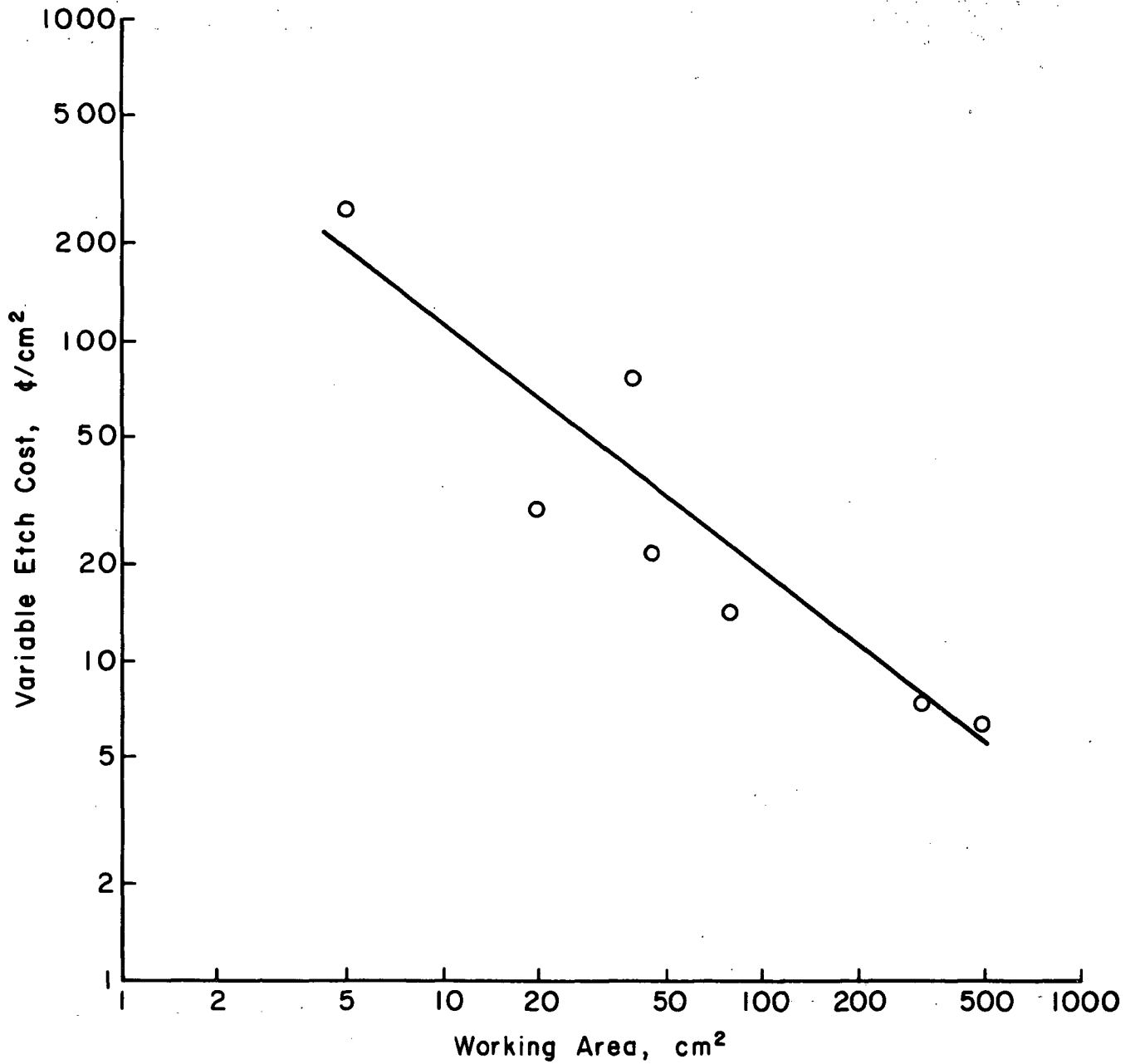


Fig. 6-5. Variable etch cost as a function of ion beam working area. The variable cost is the incremental cost for each additional etch dose of 1 mA-hr/cm².

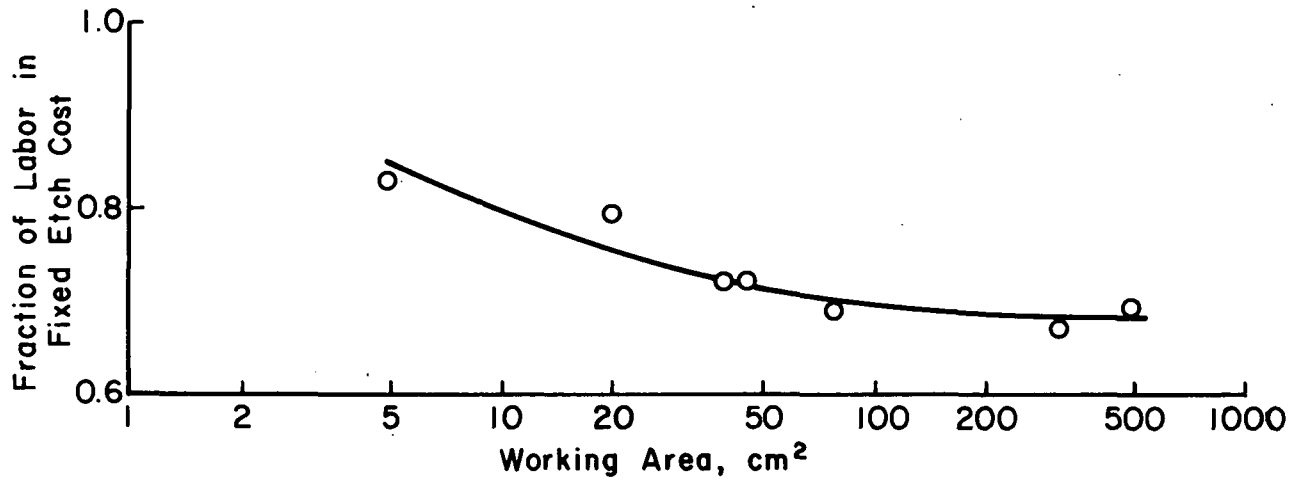


Fig. 6-6. Fraction of fixed cost that is due to labor, as a function of ion-beam working area.

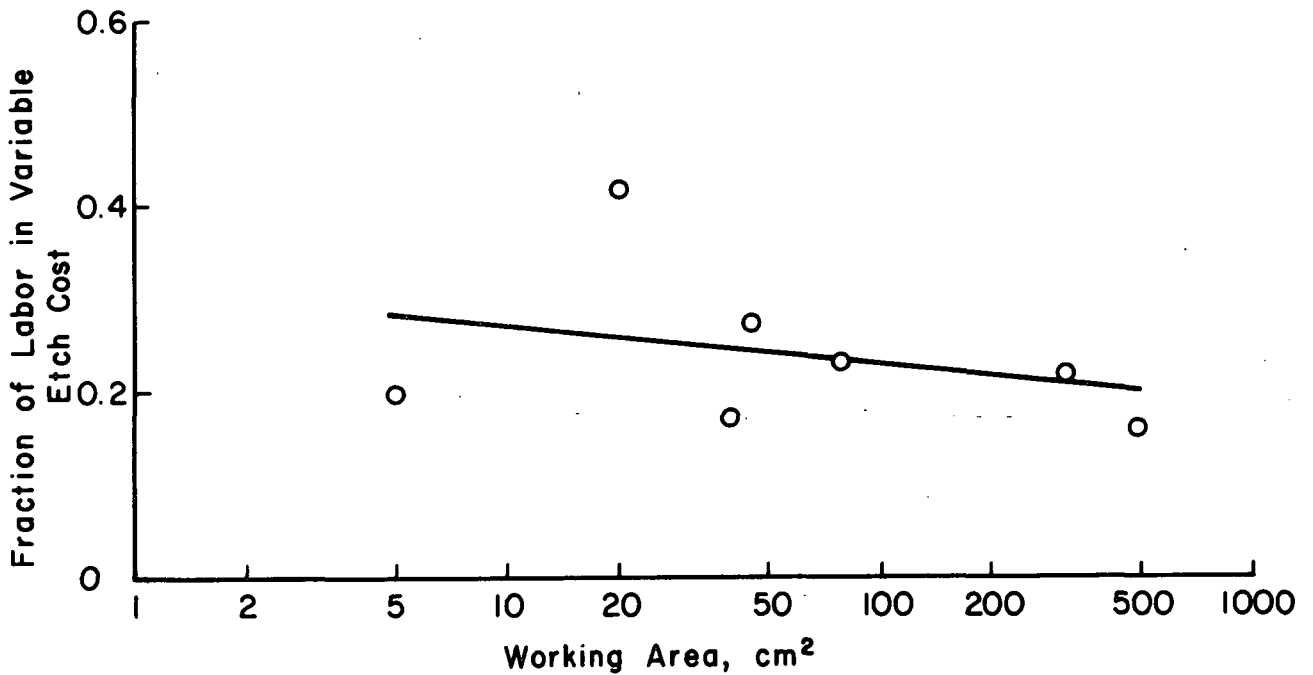


Fig. 6-7. Fraction of variable cost that is due to labor, as a function of ion-beam working area.

example, a general trend of lower costs for machines with higher ion-beam current densities.

The total etch costs (fixed plus variable costs) are shown in Fig. 6-8 as a function of the annual production rate for several etch doses. The annual production rate in cm^2/yr was obtained by fully utilizing both the working area and the 1000 hr/yr machine operating schedule. As shown, the etch cost varies more with changes in annual production rate than changes in etch dose. This apparent contradiction of an additional etch dose not affecting cost is the result of the steep decreases in costs with increasing working area shown in Figs. 6-4 and 6-5. To maintain a constant annual production with an increase in etch dose requires an increase in working area. This increase in working area results in a decrease in overall costs that roughly cancels the increased incremental cost due to the increased dose.

Future Increases in Machine Size

The preceding costing is limited to existing machines and technology. The costs show a steep decrease for an increase in working area. An obvious question is whether or not a continued cost decrease would be expected for a further increase in machine size. For this further increase in machine size, substrate handling mechanisms similar to those used in vapor deposition were assumed to increase the batch size without increasing the ion-beam working area. For an estimate of machine cost, the price of a complete vapor deposition machine was added to the price of a large ion source plus electronic console.

Using the same model for costing, the fixed cost dropped by about a factor of 3 for an increase in batch size of a factor of 10, while the variable cost remained about the same. The actual fixed cost would probably be reduced by more than a factor of 3. This is because the use of the same costing model resulted in an unrealistically high labor

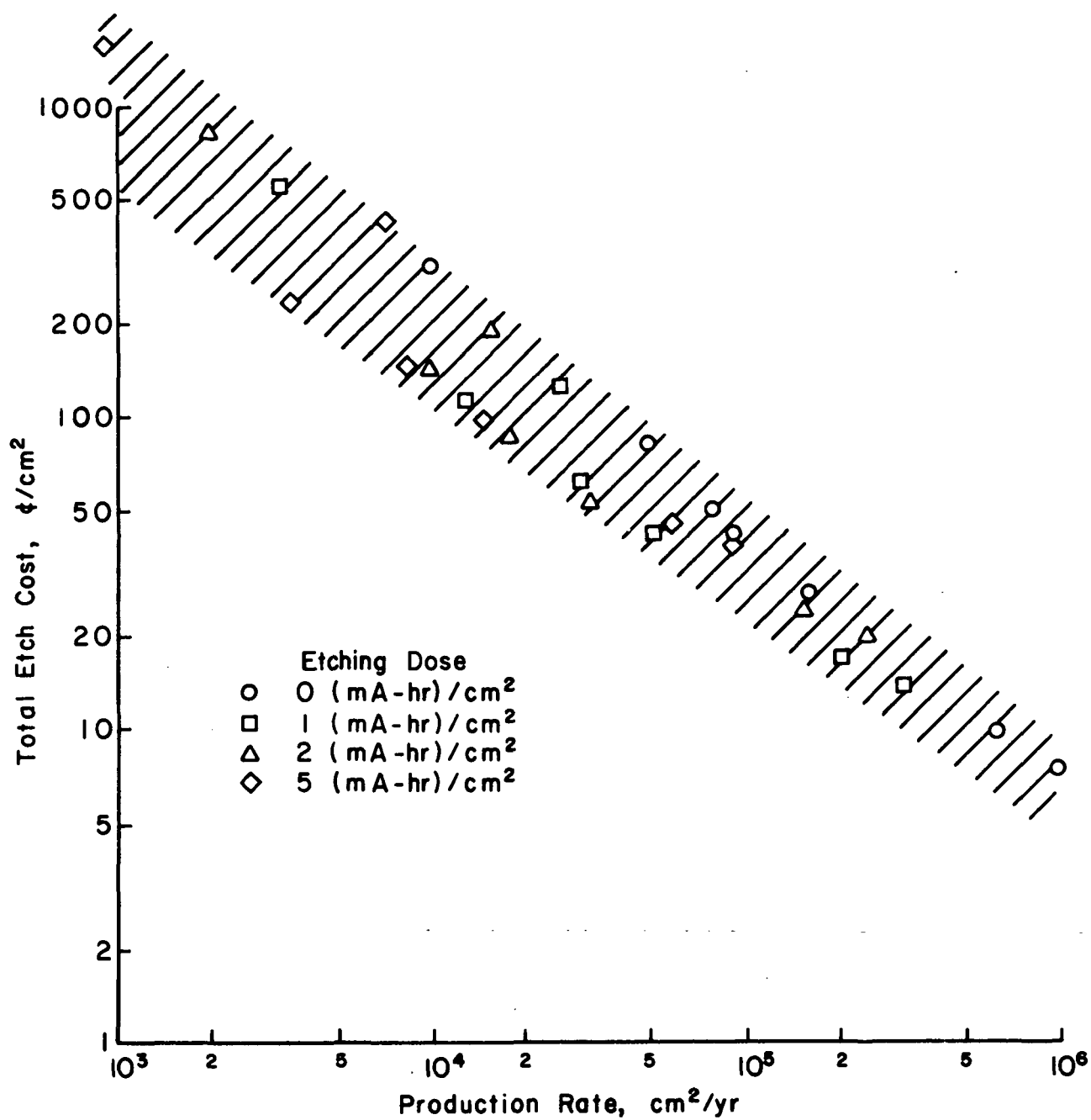


Fig. 6-8. Total etch cost as a function of annual production rate for several etch doses.

of more than 5 hours per run. The lack of any decrease in variable cost is probably due to the use of a working area obtainable with existing machines.

Another approach to obtaining larger machines would be the use of continuous feed through the working area of the ion beam instead of simply using a larger batch. Although this approach was recognized, it was felt to be beyond the scope of this costing study.

Deposition Costs

The preceding calculations are all directed at etch costs. It is impractical to study deposition costs to the same level of detail because fixtures for deposition are much less developed than etching fixtures, particularly for large ion beams. It is possible, however, to make a rough estimate of costs from general principles. The view factors between targets and deposition substrates limit deposition rates to about 20-25 percent of the target etch rate for circular ion beams. Uniformity considerations at the deposition substrate limits the area of the latter to roughly the target area. From the viewpoint of the material being etched and deposited, then, the deposition process is less efficient than the etching process by a factor of 4 or 5. For a given machine size, deposition would therefore be expected to have a production rate decrease and a cost increase of this same factor of 4 or 5.

Discussion

The cost calculations presented in this section are obviously sensitive to some process details that cannot be included in a general study. It is felt, however, that the results are sufficiently meaningful

to permit a few broad conclusions to be drawn.

One fundamental conclusion is almost self evident. The labor cost of 25 dollars/hr is so large compared to the machine cost of 5 to 20 dollars/hr that most machine operations must be unattended. This relatively high cost of labor was the basis for using labor only for specific required duties. It is also implied, of course, that the ion beam machines should be capable of unattended operation during as much of the operating cycle as possible. Most present ion beam machines could stand further improvement in this regard.

As a general conclusion, large ion-beam machines give substantially reduced etching costs. The trend is clear and general enough that further increases in size should give additional cost decreases. Within the size range studied, the etch cost was nearly inversely proportional to working area, hence production capacity. In production applications, this relationship means that a large machine can be operated at part capacity for roughly the same cost as a small machine operated at full capacity. The actual costs will depend on the specific machines and processes under consideration, but the option of excess production capacity at little or no cost increase should be considered.

It should be clear to those familiar with ion-beam technology that machines optimized for specific applications will have cost advantages over general purpose machines. Further, the combination of optimization for special applications with the economy of larger sizes should result in sharply reduced further costs. One of the application distinctions to make is in the run length. From the rough equivalence of fixed cost (Fig. 6-4) and the incremental (variable) cost for an etch dose of 1 mA-hr/cm² (Fig. 6-5), it is possible to define short and long runs.

If the etch dose is a small fraction of 1 mA-hr/cm^2 , then the run is short and the cost is primarily in labor. If the etch dose is several mA-hr/cm^2 , then the run is long and the cost is mostly in the machine.

Most etching of solid-state electronic circuits consists of short runs. For such runs the maintenance and substrate handling procedures are critical for control of the dominant labor cost. An example of a configuration that employs some labor reducing concepts is indicated in Fig. 6-9. The substrate holder is loaded outside the facility. The actual loading of the machine thus consists of removing the previous substrate holder and inserting the new holder. A major cleanliness and maintenance problem is the control of sputtered layers that accumulate on and eventually peel from facility surfaces outside of the beam area and usually after exposure to atmosphere. In the proposed configuration the substrate holder is located so that no peeled layers can fall upon the substrates. Further, with the substrate holder cleaned by the ion beam, no peeled layers can fall upon the ion source. Material falling from the cylindrical wall can accumulate for routine cleaning at the bottom of the vacuum chamber. Note that the vacuum pump port is at the side to prevent peeled layers from falling into the vacuum pump. Maintenance labor can also be reduced by providing access to the ion-source discharge chamber without removal and replacement of screws. In the configuration of Fig. 6-9, maintenance of the ion source is accomplished by raising the cylindrical section of the vacuum chamber. Access to the main cathode is by lifting the accelerator system, which is held in place only by gravity. The general approach shown in Fig. 6-9 is thus seen to have advantages for minimizing the required labor while maintaining a clean environment. The usual arrangement of ion source and substrates

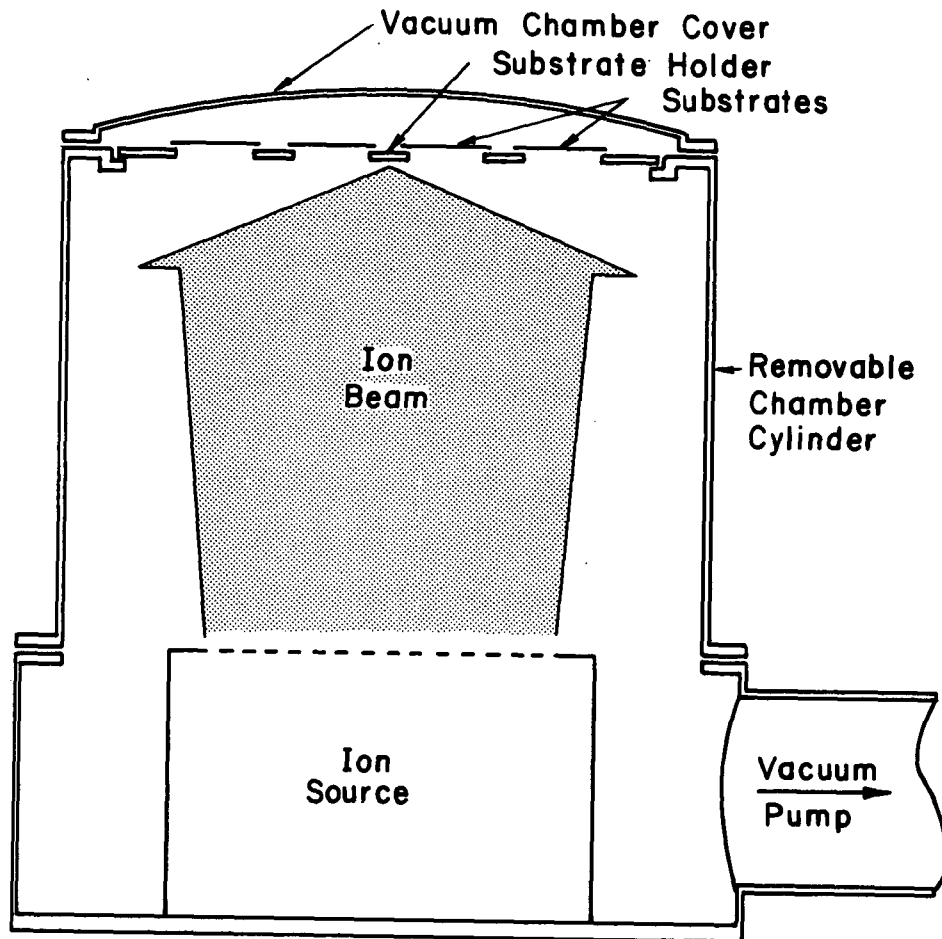


Fig. 6-9. Configuration of ion-beam machine to reduce required labor cost. This configuration should be most advantageous for short runs.

is the reverse, though, so that frequent and careful cleaning of the ion source is required to prevent peeled material falling from the source onto the substrates.

Another consideration for short runs is that a large ion-beam working area is more important than a high current density. In fact, a low current density may be advantageous because it can reduce the labor associated with heat-sinking.

Substrate handling techniques to increase the batch size were discussed in "Future Increases in Machine Size". Such techniques can be thought of as ways to effectively increase the working area without increasing the total beam current. It should be readily apparent that short runs will benefit more from such substrate handling techniques. You may recall that the cost estimates of that section showed a decrease in fixed costs, but no decrease in variable cost.

Some examples of applications requiring long runs are the deep etching of medical-implant surfaces, etching of materials with low sputter yields, and many deposition processes. All of these can require etch doses of a few mA-hr/cm². The machine cost will dominate such runs and the best way to control costs for those runs is to use a machine that is optimized for high current density in the ion beam.

Machine optimization for deposition presents special design problems that have mostly been ignored in present ion-beam machines. Ion-beam uniformity at the target is of little importance in the usual deposition application. But uniformity is critical in most etch applications, and normally requires design compromises to be achieved. An accelerator-system optimized for etch applications is therefore unlikely to be near optimum for deposition applications. Even the usual circular beam shape

is nonoptimum for many deposition applications. A narrow rectangular beam shape would, for example, permit deposition fixtures in which higher fractions of the sputtered target material could be utilized as uniform layers on the deposition substrates. If the deposition substrates were moved relative to the fixture, the fraction utilized could be increased even more.

In conclusion, the cost estimates presented in this section should be considered as only preliminary. More detailed studies can be conducted for specific applications, and should give more reliable cost estimates for those applications than the general study presented herein. The costs presented herein are also preliminary from the viewpoint of machine optimization for specific applications. The required ion source technology is available for a much higher degree of optimization than is utilized at present.⁶ The costs should decrease sharply as the available technology is more fully utilized for specific applications.

VII. STATUS OF ION-SOURCE TECHNOLOGY FOR SPUTTERING APPLICATIONS*

Broad-beam ion sources are used in a variety of sputter etch and sputter deposition applications. As indicated by the schematic diagram shown in Fig.7-1, a broad beam is obtained by using a multiaperture accelerator system, which permits much higher beam currents than would be possible with a single aperture system at the same voltages. The production of ions in broad-beam sources is almost exclusively by electron bombardment of neutral gas atoms, with the electrons provided directly or indirectly by thermionic emission. This combination was originally developed for electric space propulsion and is preferred for most ground applications because it gives efficient ionization of any element that is easily vaporized, together with good stability and control of the ionization process. A compound may also be ionized in this manner, but the compound is usually fragmented by the electron bombardment. The choice of a compound over an element is therefore usually due to material handling considerations.

The usual alternative to a broad-beam ion source for sputtering applications is the conventional plasma diode. The broad-beam ion source has several important advantages relative to this alternative choice. The energy, direction, and current density of the neutralized ion beam can be independently controlled over wide ranges. The pressure of the etching

*This section is essentially the same as an invited paper presented at the November 1977 meeting of the American Vacuum Society in Boston, which will also be included in the J. of Vac. Science and Tech., both under the title "Technology of Ion Beam Sources Used in Sputtering".

or deposition region is also substantially independent of discharge-chamber requirements. All of these advantages are associated with spatial and electrical isolation of the ion production region from the target and/or substrate, and result in much better definition and control of the sputtering environment.

This paper describes the technology involved in the ion production, ion acceleration, and electron emission processes, as well as the characteristics of the ion beam that is generated. The technology description includes present performance and future expectations.

Ion Production

Ions are produced in the discharge chamber (see Fig.7-1). Energetic electrons, usually 30 to 50 eV, are emitted from the discharge chamber, or main, cathode. Cathodes will be discussed later, but a thermionic cathode can be assumed for this section. A magnetic field between the anode and cathode prevents the premature escape of energetic electrons, thereby making possible efficient operation at low discharge-chamber pressures. The ions that are produced recombine on the discharge-chamber walls, except for the 10 to 30 percent that reaches the accelerator system and is accelerated to become the ion beam.

The discharge-chamber performance is measured in terms of the required neutral gas flow, the ion beam profile obtained, and the energy or power lost in generating beam ions. The neutral gas loss has been studied and, given the discharge-chamber and accelerator-system geometries, can be predicted.¹ To give a somewhat simplified description of the neutral-loss theory, the gas flow to a discharge chamber must be sufficient to supply the beam current plus maintain the discharge chamber in the mid- 10^{-4}

Torr range. An increase in gas flow from this value will usually result in little change in beam current, but a decrease will result in a decrease in beam current that can only be partially offset by an increase in discharge power. The ion-beam profile at the source should be as flat as possible, even in those cases where the ion-beam uniformity at the target is not important. This is because minimum mutual contamination of the ion source and the sputtering target requires maximum beam collimation to obtain maximum distance between the two. This maximum collimation is obtained with uniform current density at the accelerator system.* The most frequently used index of ion-beam uniformity is the average-to-peak current-density ratio, which varies from about 0.4 to 0.9. The discharge loss is usually not important in ground applications, but typically ranges from 200 to 1000 eV/beam ion (or watts/beam ampere).

Historically, improvements in discharge-chamber performance have resulted primarily from improved magnetic field configurations. The first broad-beam ion sources used a nearly uniform magnetic field oriented parallel to the axis of the discharge chamber.^{2,3} It was then determined that a decreasing magnetic field strength towards the accelerator system (called a divergent field design) gave lower discharge losses than a uniform field.⁴ The divergent field design is indicated in Fig.7-1, together with a typical ion-beam profile. This design was developed for electric space propulsion, where reduction of discharge losses is more important than beam uniformity. For sputtering applications, though, beam uniformity is usually more important.

*There is the alternative of varying the aperture size and the grid spacing to match the current-density profile of the ions. This approach requires an expensive and complicated accelerator system, which is usually correctly matched to the ion-beam profile at only one operating condition.

The peaked beam profile in Fig.7-1 results from electrons being emitted near the chamber axis and crossing magnetic field lines by energy absorbing collisions. The energetic electrons are thus concentrated near the axis of the discharge chamber, leading to a similar nonuniformity of ion production and extracted ion-beam current density. This nonuniformity of the ion beam is an inherent characteristic of the divergent field design, with the nonuniformity becoming more serious as the chamber diameter is increased. The average-to-peak current-density ratio ranges from about 0.4 to 0.6. Higher values of this ratio require design or operating compromises.

For ion beam diameters of 2 or 3 cm (at the accelerator system), it is easy to use a larger ion source diameter and extract the ion beam from the more uniform central portion. This "masking down" of the accelerator system increases the required discharge power for a given beam current, but the amount of power increase is usually not significant for small ion sources. For larger sizes, using an ion source that is much larger than the beam diameter is usually impractical. Decreasing the magnetic field strength will give a more uniform distribution of energetic electrons, but it will also result in a greater loss of these electrons to the anode, which increases the discharge losses. The effect of reducing field strength also depends on the specific operating conditions. Multiple cathodes can be used to make the profile more uniform, but beam surveys near a divergent-field ion source with multiple cathodes will show a nonuniformity associated with cathode placement unless a large number of cathodes is used.⁵

Later discharge-chamber development gave rise to the strongly divergent field,⁶ the radial field,⁷ and the cusped field,⁸ all of which have

improved beam uniformity over the divergent field design. These designs were developed for electric space propulsion and have not been used in sputtering applications, except where ion source hardware was available from the space program.

Multipole designs have very low magnetic field strengths except near the anode or anodes.^{9,10} As indicated in Fig.7-2, this field distribution permits the energetic electrons to have free access to most of the discharge-chamber volume, thereby producing a very uniform beam. Average-to-peak current-density ratios are about 0.9. This value is the highest ratio observed with broad-beam ion sources, and is approached only by the values obtained with radial and cusped field designs. Because the beam uniformity is inherent in the multipole design, performance compromises (such as reduced field strength) are not necessary. The multipole design is also well suited to larger sizes. A 30-cm source of this type has demonstrated excellent uniformity.¹¹

Ion Acceleration

The acceleration of ions is accomplished between an upstream, or screen, grid and a downstream, or accelerator, grid. A grounded third grid farther downstream - the decelerator grid - may, or may not, be used. These two or three grids together make up the accelerator system. The screen grid is at, or near, cathode potential, and serves to direct the ions into small beamlets. These beamlets are accelerated by the negative potential of the accelerator grid. Due to the alignment of the apertures in two grids, the beamlets escape the accelerator system to form the composite ion beam. Because of the high density of ions in this beam, the addition of electrons is normally required for charge neutralization.

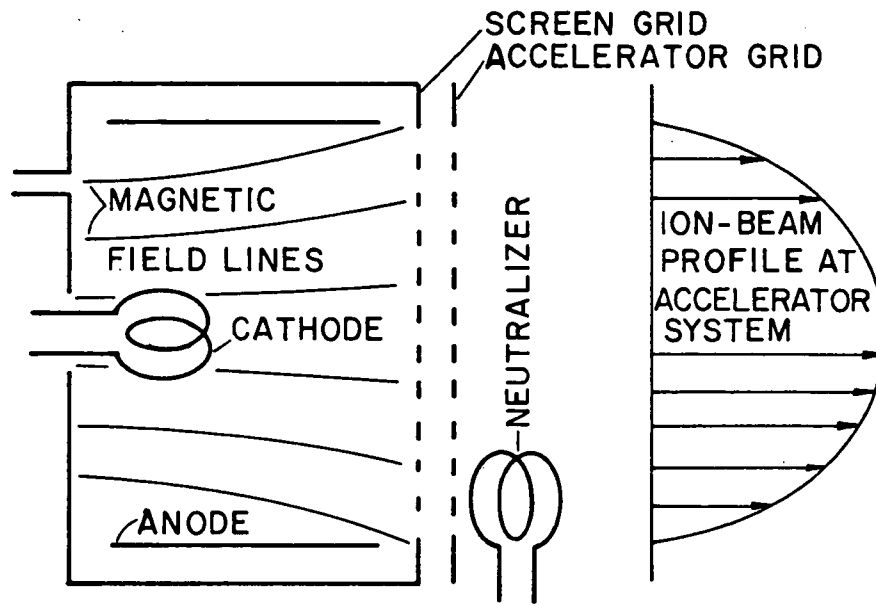


Fig. 7-1. Divergent field ion source.

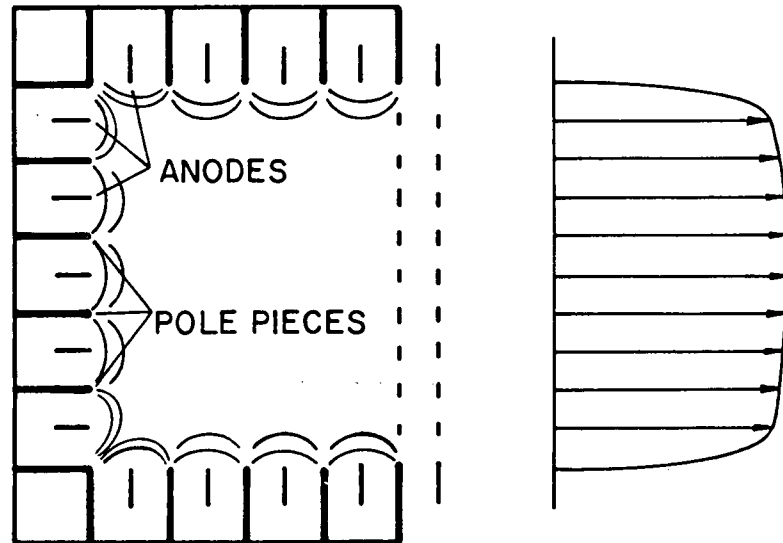


Fig. 7-2. Multipole ion source.

The recombination of electrons and ions is usually not a significant process.

The potential variation through an accelerator system is indicated in Fig.7-3. The ions originate at plasma potential, which is within several volts of anode potential. They are accelerated through the total voltage difference, V_t , between this plasma potential and the accelerator grid potential. They are then decelerated to near ground (facility) potential in the ion beam, giving a net voltage difference, V_n , from their original potential. A voltage ratio, V_n/V_t , at least slightly less than unity is desirable to provide a barrier for the neutralizing electrons.

The current capacity of an accelerator system can be obtained by solving Poisson's equation. For parallel plates, the maximum current density is given (in SI units) by Child's law,

$$j = (4\epsilon_0/9)(q/m)^{1/2} V^{3/2}/\ell^2 \quad (1)$$

where ϵ_0 is the permittivity of free space, q/m is the charge-to-mass ratio for the accelerated particles, V is the potential difference between the two plates, and ℓ is the distance between them. The constant $4\epsilon_0/9$ will change for different electrode configurations, but the rest of the Eq. (1) will be unchanged. For a broad-beam accelerator system, V should be replaced by V_t and ℓ replaced by approximately the spacing between grids ℓ_g . With these substitutions, Eq. (1) can be rearranged to give

$$j/V_t^{3/2} \cong K(q/m)^{1/2}/\ell_g^2, \quad (2)$$

where K is a constant depending on the particular electrode configuration. Maximum current capacity at a given voltage thus corresponds to a maximum value of perveance, $j/V_t^{3/2}$. An accelerator system will usually operate over a wide range of perveance below this maximum value.

The collimation of an ion beam is measured in terms of a beam spread angle, which is determined downstream of the neutralization plane where the ions follow essentially straight trajectories. The typical variation of beam spread with perveance is shown in Fig. 7-4 for a two-grid system operating at several voltage ratios.* The maximum perveance is approximately the same for all values of V_n/V_t , and corresponds to direct impingement of the beamlets on the accelerator grid. The minimum beam spread is usually found at a perveance less than the maximum value, as indicated in Fig. 7-4. As also indicated, the minimum beam spread is smaller for larger values of V_n/V_t .

Most sputtering applications require a high current density and good collimation, as well as operation at or below some maximum V_n . The high current density reduces the time for the sputtering operation. Good collimation permits a large distance between the sputtering target and the ion source, thus reducing mutual contamination with sputtering particles. The limit in V_n is associated with target damage, target heating, or the variation of sputtering yield with energy. Inasmuch as current density increases with V_n for constant V_n/V_t , the usual tendency is to operate at as high a V_n as permitted. Assuming operation at some maximum V_n , the collimation and current-density requirements are contradictory.

*The qualitative trends shown in Figs. 7-4 and 7-5 can be obtained from a study that includes over 300 digital computer solutions of two and three grid systems.¹²

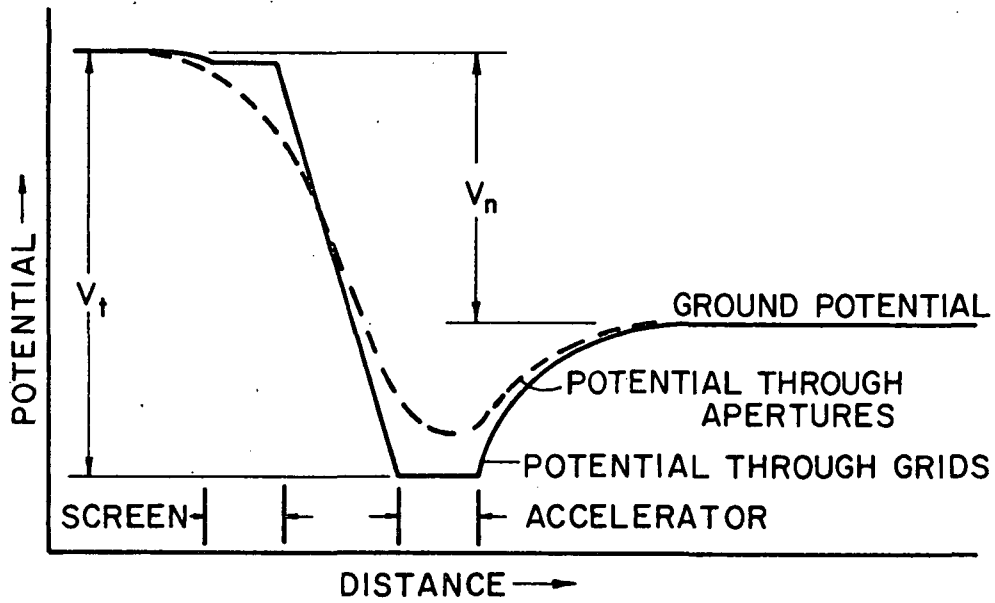


Fig. 7-3. Variation of potential through an accelerator system.

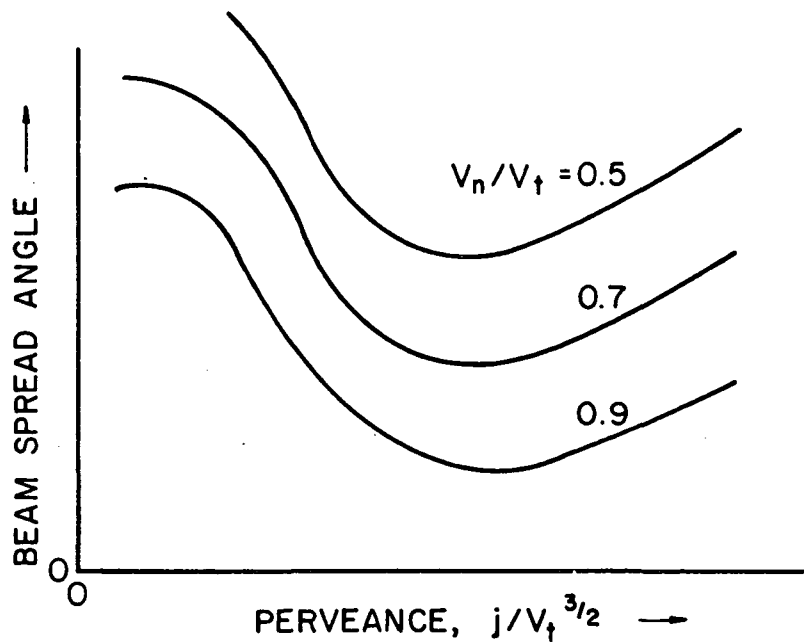


Fig. 7-4. Variation of beam spread with perveance for two-grid accelerator system.

High current density requires low V_n/V_t (high V_t), while good collimation requires high V_n/V_t .

Figure 7-4 is for a two-grid accelerator system. The addition of a third (grounded) grid changes the problem somewhat, as indicated in Fig. 7-5. With the third grid, the beam divergence is decreased at low values of V_n/V_t . But at high values of V_n/V_t , where the smallest beam divergence is found, the third grid gives no reduction in beam divergence. The value of a third grid, then, is to reduce the beam divergence when a low value of V_n/V_t is used to increase beam current density. If sufficient current density is available at a high V_n/V_t , then the beam divergence is already minimized and a third grid has no advantage.

An important parameter that was left out of the preceding general discussion is the grid spacing ℓ_g . From Eq. (2), the current density can be increased by reducing ℓ_g . In some cases, electrical breakdown limits the minimum ℓ_g . The usual case, though, is that the minimum spacing is determined by thermal distortion or warping. The first type of accelerator system that was widely used was constructed of flat molybdenum sheets. This approach permitted beam diameters up to about 60 times the grid spacing.¹³ The choice of molybdenum was based on low thermal expansion, low sputtering yield, and good mechanical strength. The latter was required for the launch environment. Tests were also made with carbon grids as part of the space propulsion program.¹⁴ Carbon showed greater resistance to warping, but had insufficient strength for the launch environment. Strength is not as important in ground applications where the source remains stationary and carbon can be used for larger ratios of beam diameter to grid spacing than are possible with flat molybdenum grids.⁹ The largest ratios of beam diameter to grid

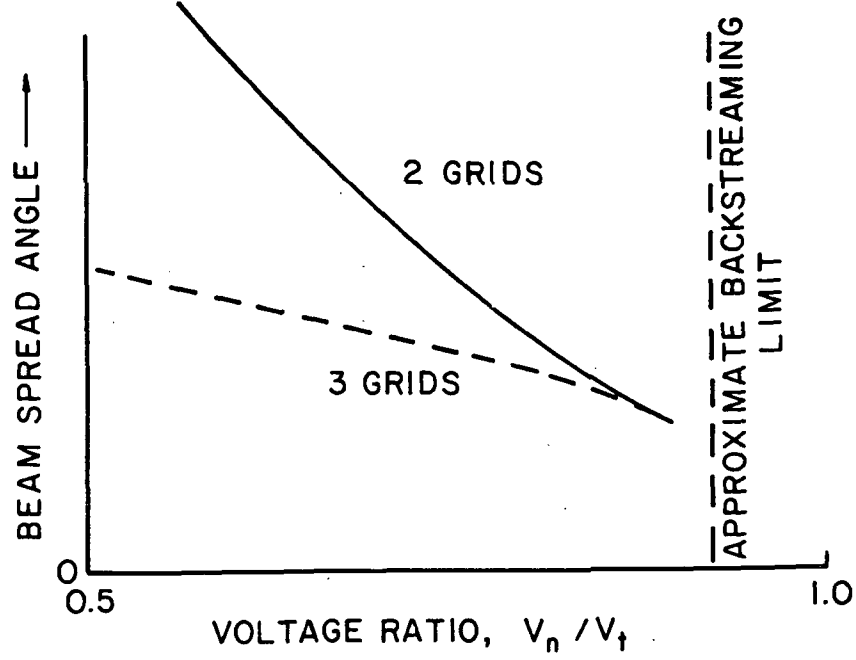


Fig. 7-5. Comparison of beam spread for two-grid and three-grid accelerator systems at constant perveance.

spacing are obtained with dished molybdenum grids, which are also a product of the space propulsion program.¹⁵

The proper approach to grid design is to use whatever level of technology is needed to give the required ratio of beam diameter to grid spacing, moving from flat molybdenum to carbon to dished molybdenum as the required ratio increases. Dished molybdenum has been used in sputtering ion sources with 30-cm beams. In this size, there was no problem reaching 1 mA/cm^2 with well collimated 500 eV argon ions.¹¹

Interelectrode supports (within the beam area) were subjected to considerable study as part of the space program.¹⁶⁻¹⁸ Such supports, if successful, would permit larger beam diameters with the same grid spacing. The general conclusion of this study was that interelectrode supports would not be reliable without a large local blockage of the accelerator system, hence causing considerable loss in beam current. The adverse effects on beam uniformity are probably more important than beam current losses for ground applications.

The current trend in accelerator system technology is toward specific designs for specific applications. Two types of designs have been used enough times that it is clear they will survive as design categories. One is low voltage etching. An example of this category is 2 mA/cm^2 over a 10 cm beam diameter (at the accelerator system) with 500 eV argon ions. Uniformity is important for this category, and the current density is constant within $\pm 5\%$ over the center 5 cm of diameter at a distance of 15 cm from the source.⁹ The other category is sputter deposition, for which the example is 4.5 mA/cm^2 over a 5 cm diameter (at the accelerator system) with 1300 eV argon ions.¹⁹ Low deposition pressure is important for much deposition work, and this small beam diameter reduces the loss

rate of neutrals from the ion source. Both of these examples operate at high ratios of V_n/V_t and therefore have excellent collimation. Both of these examples are also used on the same 10 cm ion source. The reason for specialized designs is clear when we exchange the uses of these two examples. If the low voltage design were used at 1300 eV, it would have excessive neutral losses. It might also not work due to excessive arcing, which would result from the close grid spacing that is used. On the other hand, the high voltage design would have only a small useful area when used for 500 eV etching. It would also have a current density of only 1 mA/cm^2 if used with a high value of V_n/V_t .

There are several other specialized designs that are each being used by only one or two researchers. It is not clear at this point which of these other designs will be widely used in the future. There are enough designs being evaluated, however, that it is clear that some will find their way into general use. It is also clear that the performance advantages of specialized designs are too large to ignore.

Electron Emission

Ribbons and wires of both tungsten and tantalum have been used for electron emitters, although wires have been used more frequently. These cathodes usually give lifetimes of a few tens of hours in the discharge chamber and a few hours as a neutralizer. By using sufficiently heavy cathodes and high enough heating powers, these lifetimes can be extended by up to a factor of 10. These lifetimes also assume the use of argon or another inert gas. The presence of reactive materials, such as oxygen, can drastically reduce the lifetimes.

The relatively short lifetime of the neutralizer cathode is the result of sputtering by beam ions (see Fig. 7-1). Placing the neutralizer

outside the ion beam would avoid the direct sputtering, but would result in excessive beam potentials (due to poorer coupling) and therefore increased sputtering by charge-exchange ions. The external location for a thermionic neutralizer cathode appears practical only for beams that are several cm, or less, in diameter and operating at 1000 eV or more.

A large number of other cathode types were studied as part of the space propulsion program, but probably only one of these is of interest for ground applications. This is the hollow cathode type in which a gas discharge through a small orifice provides much of the electron emission. In the hollow cathode developed for space propulsion, the operating characteristics are controlled by a gas flow through the orifice. Hollow cathodes were the subject of considerable development for space propulsion with vaporized mercury and cesium as the gas. The application of this hollow cathode technology to ground applications, however, has not been as straightforward as similar applications of discharge-chamber and accelerator-system technologies. This has been partly due to the different cathode characteristics when operated with argon. It has also been the result of the construction used for space applications, with permanently installed inserts that are either coated or impregnated with barium and strontium oxides. The configurations used were well suited to occasional exposure to atmosphere, but were not suited to repetitive exposures day after day.

A modification of the hollow cathode was developed for controlled internal electron emission in space propulsion research. This modification uses an internal thermionic emitter in place of the oxide coated or impregnated insert.²⁰ This modification has recently been adapted for neutralizer use in ground applications.²¹ Because the hollow cathode

generated a plasma plume, it is possible for the neutralizer to have excellent electrical coupling to the beam with the neutralizer hardware well outside the beam. Inasmuch as an immersed neutralizer emitter is usually the largest contributor of sputtering contamination from the ion source, the use of a hollow cathode has obvious advantages for high purity sputter deposition. The lifetime of the internal emitter is about the same as that of the discharge-chamber cathode when argon is used. When reactive gases are used in the ion source, they do not affect neutralizer emitter lifetime if an inert gas flow is used through the neutralizer.

An obvious future improvement is the use of the hollow cathode for the discharge chamber. Such use, together with a hollow cathode neutralizer, would remove most of the cathode lifetime limits associated with reactive gases.

Ion Beam Characteristics

The ion beam effectively originates at the potential of the plasma within the discharge chamber. This plasma is nearly uniform in potential, so that the associated energy spread in the ion beam is only several eV. The plasma is usually within about 5 volts of the anode potential. The discharge-chamber anode is therefore the hardware element that is closest to the source potential for ions. The fraction of doubly charged ions is kept under 1 or 2 percent by using a low enough discharge voltage (~ 40 for argon). From past experience with mercury, the fraction of doubly charged ions will tend to rise with increases in discharge-chamber size. The theory describing this relationship with argon is

being studied at present and should be available in the near future.*

The neutralizing electrons in an ion beam have a nearly Maxwellian distribution. Due to the good thermal conductivity of the electron gas, the electron temperature is nearly constant throughout the ion beam. This temperature is usually 1 to 2 eV, with the higher temperatures usually associated with lower ambient pressures. The electron density varies in accord with the Boltzmann relation,

$$n_e = n_o \exp(qV/kT), \quad (3)$$

where the reference electron density n_o is at the plasma potential V equal to zero. Equation (3) has important implications for neutralizer theory. The local electron and ion densities are essentially equal because the Debye distance is much smaller than the ion beam dimensions. A variation in ion density will, through Eq. 3, result in a corresponding variation in plasma potential. A grounded probe that is receiving no net current in one ion beam location will therefore indicate a net current if moved to a region of different ion density. A related problem is that the electron current picked up by a grounded probe will vary drastically with small changes in beam potential. The net result of these considerations is that substantial departures from zero current to a grounded probe should be accepted without much concern about ion trajectory deflection. The only exceptions appear to be the fabrication of gated semiconductor devices, which can be very sensitive to trapped charge.

*This theory has since been published in CR-135226.

Conclusions

There is a major advantage that broad-beam ion sources have over competitive microfabrication techniques such as the conventional diode configurations. The processes of electron production, ionization, and acceleration of ions are all independently controlled in the usual broad-beam source. It is therefore practical to optimize all these processes for a single application. The development of optimized broad-beam sources for specific future applications should therefore be much easier than the corresponding development that has already taken place in competitive techniques. The development of specialized accelerator systems will probably be the most evident aspect of this optimization.

VIII. CONCLUDING REMARKS

Plasma probe surveys in the 30-cm discharge chamber verify that the excellent uniformity in the ion beam is the result of a corresponding uniformity in the discharge chamber. The general design approach used to obtain uniformity, a flat multipole discharge chamber with a continuous cathode near the outside wall, is also justified by the probe surveys.

The design, fabrication, and subsequent use of the 15-cm multipole sputtering source was an additional and successful example of multipole technology use.

The preliminary survey of texturing included several seed materials and a variety of surface materials. The etch doses used with argon ranged from 200 to 240 mA-min/cm², while those with tetrafluoromethane (CF₄) ranged from 20 to 24 mA-min/cm². Despite the much smaller etch doses used with CF₄, deeper texturing was often obtained with CF₄. In particular, the texturing of stainless steel with CF₄ appears of possible interest for medical implants.

The interaction of a beam of argon ions passing through a background gas of argon is of interest for industrial applications of broad-beam ion sources, particularly for the etching applications. There are two dominant processes in this interaction, charge-exchange collisions of argon ions with argon and elastic collisions of argon with argon. Because the cross section for charge exchange is much larger than for elastic collisions, the elastic collisions of argon ions on argon are much less important. The cross section for charge exchange has been studied previously and is available for calculations of this nature. The cross section for elastic collisions has not been available for the

energy range of interest, and has been calculated herein from argon-argon potential data. For a 10 percent loss of ions in a beam by charge exchange, for example, traversal of 1.4×10^{-3} Torr-cm is required. In comparison, a 10 percent loss of beam energy through elastic collisions requires a traversal of $25. \times 10^{-3}$ Torr-cm. It should be clear that a substantial fraction of charge exchange can take place without significantly affecting the momentum, energy, or, to a first approximation, the sputtering capability of the beam. The momentum and energy losses in the beam can also be used to determine pressure, hence pumping, requirements in etching applications. Because the necessary elastic cross sections for argon-argon collisions have not been available prior to this publication, the calculation of beam momentum and energy losses has not previously been possible.

An obvious use of data in the costing section is to estimate costs for proposed production applications of ion-beam sputtering. The costing data can also be used for indications of future ion-beam machine needs. The hourly cost of labor is greater than the hourly machine cost for all ion-beam machines considered. This relative cost of labor and machine makes evident the need to minimize attended operation in future machines. The rapid decrease in costs with increasing machine size is a clear indication of future need for even larger ion-beam machines. It is also a quantitative justification for the previous development of the large 30-cm ion source under this grant. The costing data also make clear that it is useful to separate short and long runs in ion-beam machines, with about 1 mA-hr/cm^2 in the beam being the rough dividing line. For short runs, the labor cost is dominant in the costing. This labor cost can be reduced by improving substrate handling procedures and using

large beam areas. The ion current density is much less important than the beam area for cost reduction of such runs. Most etching of solid-state electronic circuits falls in the short run category. For the long run category, the cost of the machine is dominant and designing for high ion current density is an effective approach to reducing this machine cost. Some examples of long runs are the texturing of medical-implant surfaces, many deposition processes, and etching that is particularly deep and/or involves low sputter yield materials. Because of the different requirements for short and long runs, minimum costs will require different designs for these two types of applications.

The review of ion source technology for sputtering applications emphasizes the capacity to simultaneously optimize the processes of electron production, ionization, and acceleration of ions for a single application of a broad-beam ion source. This simultaneous optimization is generally not possible in other types of sputtering machines such as DC and RF plasma diodes. The development of specialized accelerator systems for specific applications is already taking place and will probably be a major aspect of future optimization for production applications.

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